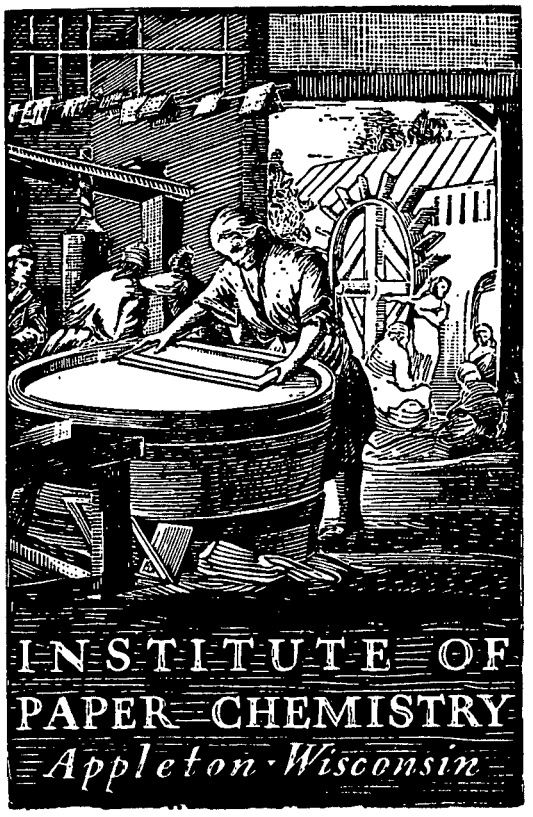

WOOD/BARK ADHESION AND METHODS OF REDUCING ADHESION IN HARDWOOD SPECIES

Project 2929

Report Two

A Progress Report

to

MEMBERS OF GROUP PROJECT 2929

June 11, 1971

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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WOOD/BARK ADHESION AND METHODS OF REDUCING ADHESION IN HARDWOOD SPECIES

SUMMARY

Using previously described field sampling techniques and the IPC Instron method of measuring wood/bark adhesion, the seasonal variation in wood/bark adhesion of sugar maple and bur oak was measured. Morphological examination of seasonal changes and differences between species in wood and bark were recorded and these observations compared with the adhesion values. The sugar maple trees sampled were slow growing and had a relatively short period of cambium activity. The bur oaks were rapid growing and had a long period of active cambium growth. Wood/bark adhesion during the dormant season and during the season of active cambium growth for oak and maple did not differ greatly from the values obtained for aspen and white birch. Zones of weakness for all four species were similar with the "cambium zone" being important during the peeling season and the inner bark region being the apparent zone of weakness during the dormant period. Because of the similarities in zones of weakness and in adhesion levels, methods successful in reducing wood/bark adhesion for one species very likely will be satisfactory for all four species under investigation.

Several approaches for reducing wood/bark adhesion, including thermal, chemical, and mechanical techniques, were investigated in a series of preliminary trials aimed at causing separation in either the cambium zone or the inner bark region near the cambium. Thermal and mechanical methods appeared to offer the most promise in the preliminary trials. The most effective thermal treatment reduced wood/bark adhesion of all four species by approximately 50%, resulted in failure in the cambium zone, and produced adhesion values comparable to midsummer sap peeling.

Plans for the program during the next six months include: (1) completion of the measurements on seasonal variation in wood/bark adhesion for slash pine, white spruce, shagbark hickory, and a southern source of eastern cottonwood; (2) completion of the morphological observations on the abovementioned species; (3) initiation of a series of additional preliminary trials on methods of reducing wood/bark adhesion.

INTRODUCTION

Research on the measurement of wood/bark adhesion and methods of reducing adhesion was initiated on March 15, 1970. The objectives of this work were to: (1) measure accurately seasonal changes in wood/bark adhesion for sugar maple, white birch, quaking aspen, white oak, shagbark hickory, white spruce, southern cottonwood, and loblolly pine; (2) examine between-species and seasonal morphological differences in an attempt to correlate morphological differences with measured wood/bark adhesion; (3) develop suitable methods of reducing wood/bark adhesion based upon information obtained regarding the causes of adhesion. Progress Report One reviews related studies, describes field collection methods and provides a detailed description of the Instron method developed for measuring wood/bark adhesion. Progress Report One also provides data on seasonal variation in wood/bark adhesion and related morphological observations for white birch and quaking aspen.

Wood/bark adhesion was also measured for bur oak and sugar maple; however, the morphological descriptions for these two species were not completed in time to be included in Progress Report One. In the report that follows, the seasonal variation in wood/bark adhesion and related morphological observations are described for bur oak and sugar maple. Also included in Progress Report Two are the results of preliminary trials aimed at reducing wood/bark adhesion.

EXPERIMENTAL METHODS AND MATERIALS

The experimental approach used consisted of periodically sampling locally grown pulpwood-sized sugar maple and bur oak from April until early November. A small chain saw was used to remove wedge-shaped samples and after trimming the samples to appropriate size, wood/bark adhesion was measured using the Instron tester.[1] The procedure used measures shear parallel to the grain in the "cambium zone."[2] The test procedure has the disadvantage that, during periods of high adhesion, failure occurs in the bark and when this happens the values obtained must be interpreted as indicating that adhesion in the "cambium zone" and between wood and bark elements immediately adjacent to the cambium zone is "in excess of the test values obtained by the testing procedure."

Upon completion of the wood/bark adhesion measurements, representative samples were embedded, thin sections prepared, the sections stained and the failure surfaces examined for seasonal and between-species differences. A detailed description of the microtechniques employed is given in Project 2929, Progress Report One.

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[1] A detailed description of field sampling procedures and the Instron testing procedure is available in Project 2929, Progress Report One.

[2] Cambium zone — the true cambium consists of a single layer of dividing cells from which the xylem (wood) and secondary phloem (inner bark) arise. In this study the term "cambium zone" has been used to designate the true cambium plus all undifferentiated xylem and phloem cells immediately adjacent to the cambium.

SEASONAL VARIATION IN WOOD/BARK ADHESION

SUGAR MAPLE

Anatomical Structure of Wood and Bark

The wood (xylem) of sugar maple (Acer saccharum Marsh.) is made up of fibers, vessels and ray cells. Sugar maple is classified as a diffuse porous wood (see Fig. 1). The vessel elements exhibit little variation in size throughout the growth rings. There are 40-80 vessels per square millimeter, occurring as solitary vessels or in multiples of two or more. The largest vessels average 75-80 μm. in diameter and between 0.4 and 0.5 mm. in length. The xylem fibers average 20-25 μm. in diameter and approximately 0.8 mm. in length. Fiber cell wall thickness averages 2-3 μm. The wood rays of sugar maple are of two types. The larger rays are unstoried, essentially homogeneous and are broad (mostly 5-7 seriate) and up to 0.8 mm. in height. The small rays are narrow (principally uniseriate) and less than 0.2 mm. in height.

The inner bark (secondary phloem) of sugar maple is composed of alternate bands of sieve tubes, phloem parenchyma, and sclerenchyma cells. These cells are bounded radially by homogeneous rays which are variable in width but principally 3 to 6 seriate and average more than 0.5 mm. in height. The small rays prominent in the xylem are only evident in the cambium zone or in the secondary phloem immediately adjacent to the cambium zone. The thin-walled sieve tubes are in 2-3 tangential rows and these uncollapsed cells in the cambial region average 35-40 μm. in tangential diameter. The mean length of the sieve tubes according to Chang (1) is 343 μm. (range 120 to 560 μm.). Sieve tubes further removed from the cambial region usually become crushed.

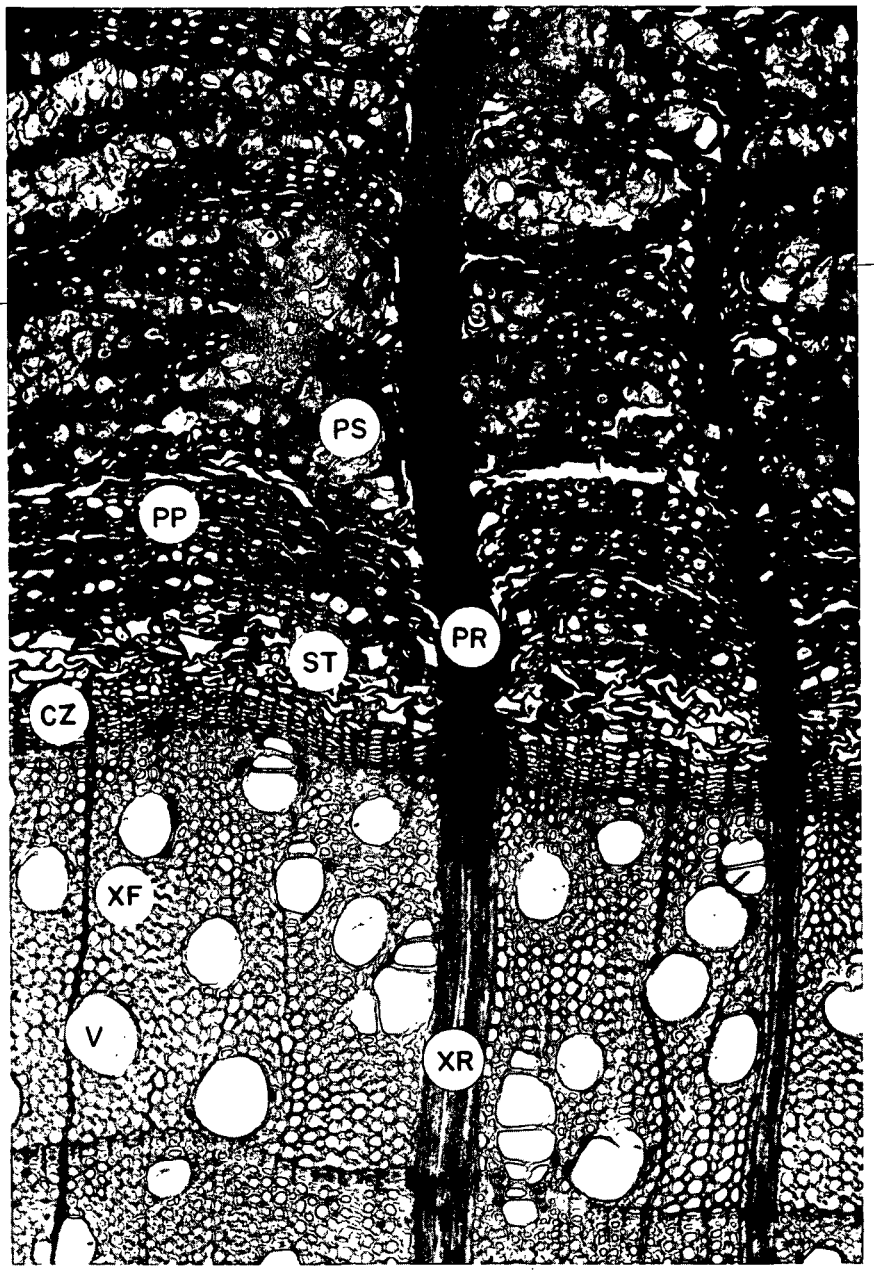

Figure 1. Cross Section of Sugar Maple Wood and Bark when the Cambium was Dormant. Illustrated are Xylem Fibers (XF), Xylem Rays (XR), Vessels (V), Sieve Tubes (ST), Phloem Rays (PR), Phloem Parenchyma (PP), Phloem Sclereids (PS) and the Cambium Zone (CZ)

The phloem parenchyma cells are distributed in tangential rows 1-3 cells in width throughout the secondary phloem. These cells average 20-25 μm. in diameter and 200 μm. in length. The cells in the outer region of the secondary phloem retain, for the most part, their original size and shape.

Tangential bands of sclerenchyma, principally groups of very thick-walled sclereids, are characteristic of the secondary phloem of maple. These bands of sclerenchyma are generally separated by 3-4 rows of parenchyma cells and crushed sieve tubes. A few discontinuous tangential bands of fiber sclerenchyma cells, which first appear in most specimens of this species in the middle of the secondary phloem, are also present in the inner bark. These thick-walled fibers have an average diameter of 14 μm. and a length of approximately 0.7 mm.

Seasonal Variation in Wood/Bark Adhesion

Seasonal sampling of sugar maple wood/bark adhesion was initiated on April 14. Measurements were continued throughout the growing season and were discontinued after the October 5 samples were tested. Table I summarizes both the morphological observations made on the test specimens and the results of the measurements taken using the previously described Instron testing procedure. Figure 2 graphically presents the seasonal variation in sugar maple wood/bark adhesion measurements as measured by shear parallel to the grain. Figure 3 illustrates the seasonal changes that occurred in the cambium zone and Fig. 4 demonstrates the accompanying changes that were found in the location of the zone of failure. Described below are the observations made on seasonal morphological changes that were associated with changes in wood/bark adhesion.

April 14 — Cambium dormant; cambium zone 6-8 cells in width; failure occurred in the last formed phloem parenchyma — sieve tube area and the

TABLE I

SUMMARY OF OBSERVATIONS ON SEASONAL VARIATION IN
SUGAR MAPLE – APPLETON, WISCONSIN

Date	Adhesion, kg./cm.^{2} Average	Adhesion, kg./cm.^{2} Standard Deviation	Cambium Activity[a]	Width Cambium Zone	New Xylem Cells Total No.	New Xylem Cells No. Non-lignified	No. Immature Phloem Cells	Location of Zone of Failure	Additional Zone of Apparent Weakness
4/14/70	10.3	0.31	D	4-5	0	0	3-4	Inner bark region, in zone of last-formed phloem parenchyma & sieve tubes and inside the last-formed bands of thick-walled sclerenchyma cells	Cambium zone
5/4/70	8.7	1.01	D	6-8	0	0	3-4	Approx. same as 4/14/70	Cambium zone
5/18/70	8.5	0.74	D	6-8	0	0	3-4	Partially in the phloem parenchyma & sieve tube area as 4/14 & 5/4 and partially in cambium zone	Cambium zone
6/1/70	5.4	0.55	A	10-12	0	0	3-4	In cambium zone – 3-4 cells outside previous year's mature xylem	Inner bark region where failure occurred 4/14/70
6/29/70	6.2	0.56	A	6-8	10-12	5-6	3-4	In inner bark, between last-formed sclerenchyma cells and bands of phloem parenchyma & sieve tubes near cambium	Cambium zone
7/22/70	7.9	1.11	D	6-8	24	6-8	6-8	Same as 6/29/70	Cambium zone
8/10/70	10.8	0.44	D	4-5	24	0	6-8	Same as 6/29/70	Cambium zone
8/24/70	10.2	0.67	D	4-5	24	0	5-6	Same as 6/29/70	Cambium zone
9/21/70	13.2	0.21	D	4-5	24	0	3-4	Same as 6/29/70	Cambium zone
10/5/70	11.6	0.44	D	4-5	24	0	3-4	Same as 6/29/70	Cambium zone

[a] A = active, D = dormant.

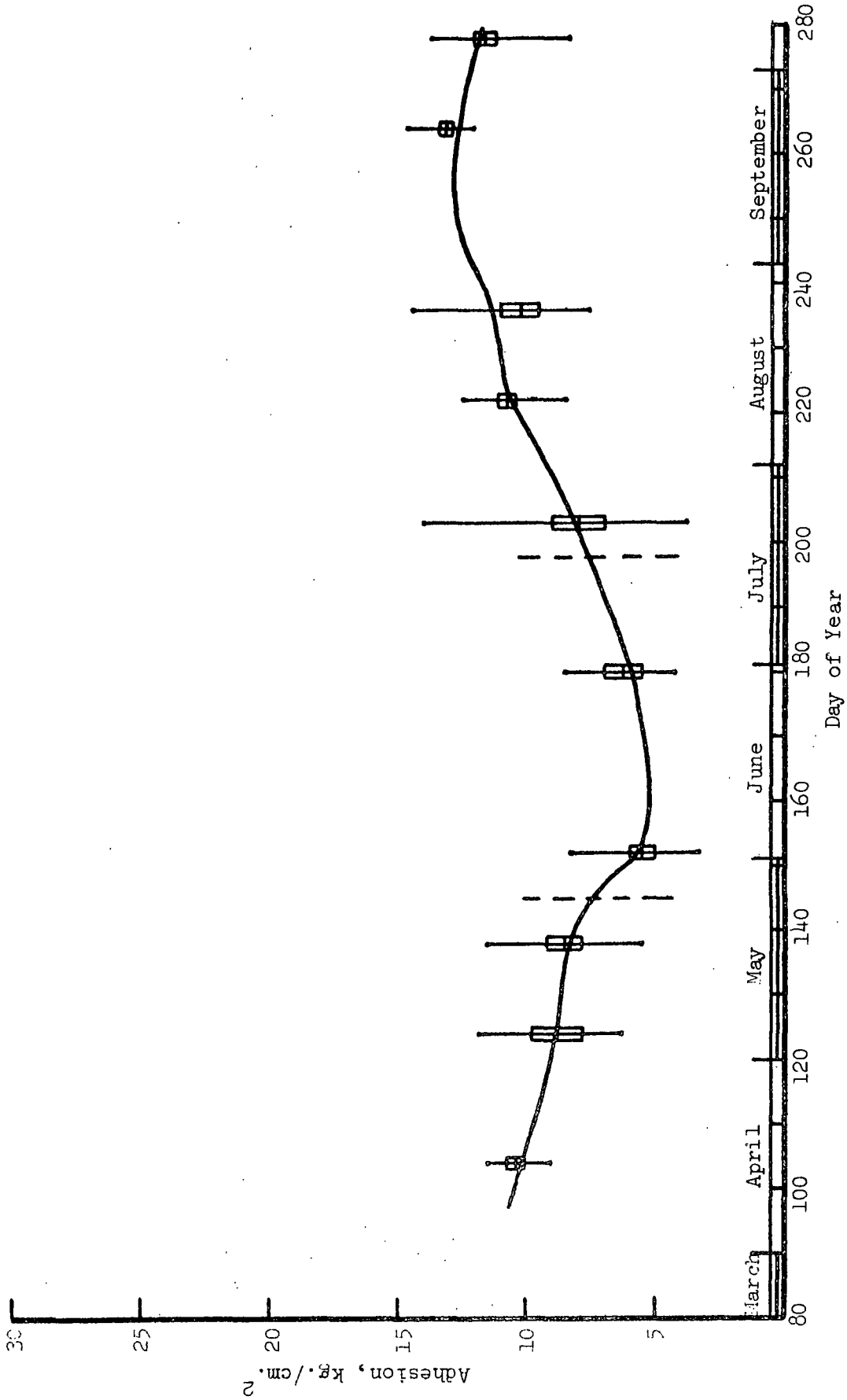

Figure 2. Seasonal Variation in Wood/Bark Adhesion for Sugar Maple. Shown for Each Sampling Date is the Range, the Mean and one Standard Deviation each Side of the Mean. The Vertical Dashed Lines Indicate the Estimated Start and End of the Peeling Season

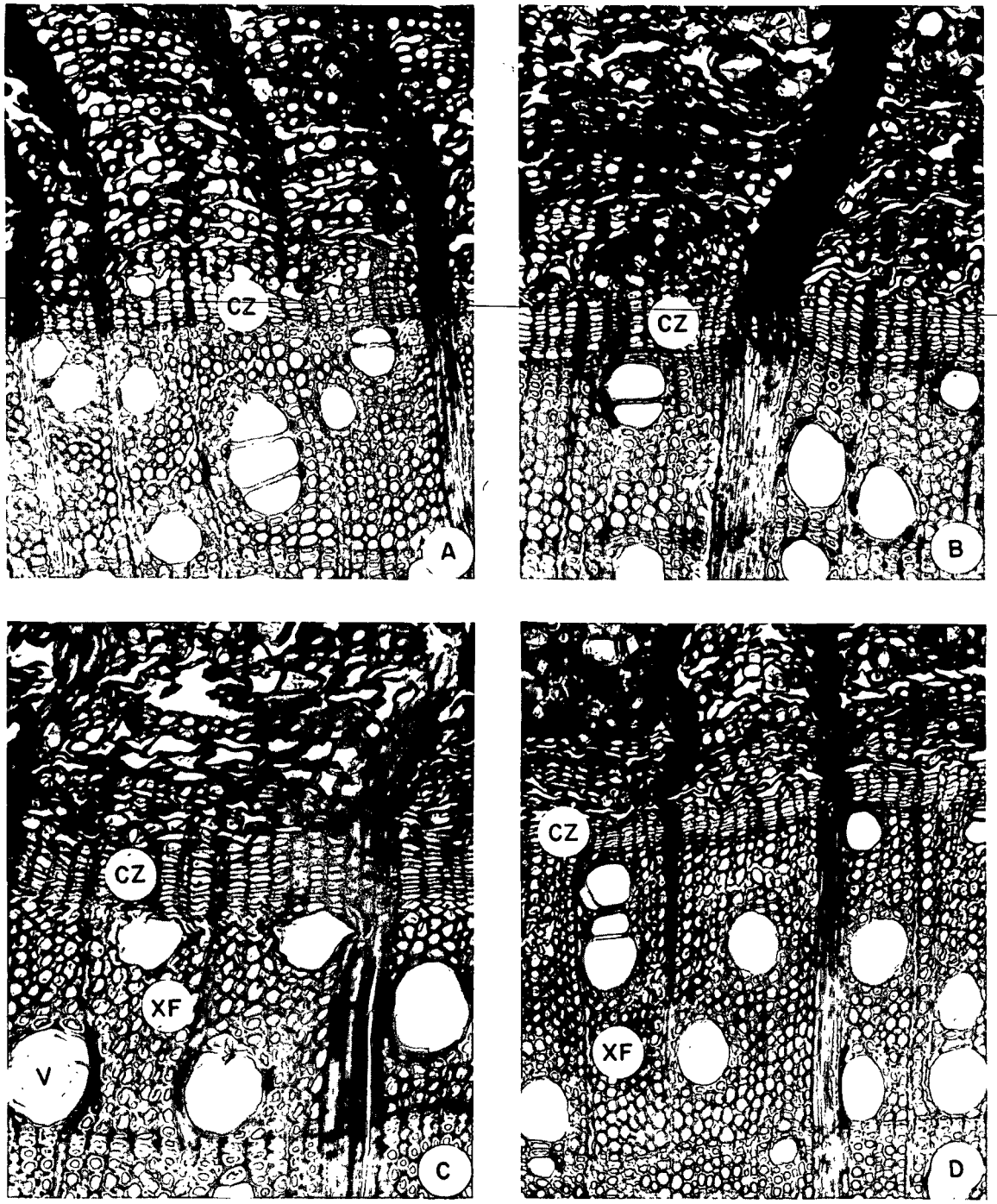

Figure 3. Illustrated, Using Cross Sections of Sugar Maple, are the Seasonal Changes that Occurred in the Cambium Zone; A — May 18 Collection, Showing Inactive Cambium Zone (CZ); B — June 1 Collection, Cambium Activity just Starting, no Newly Differentiated Xylem Cells Evident; C — June 29 Collection, Cambium Still Active, 10-12 New Rows of Xylem Fibers (XF) Present; D — August 10 Collection, Cambium Dormant, Growth and Lignification for the Year Completed

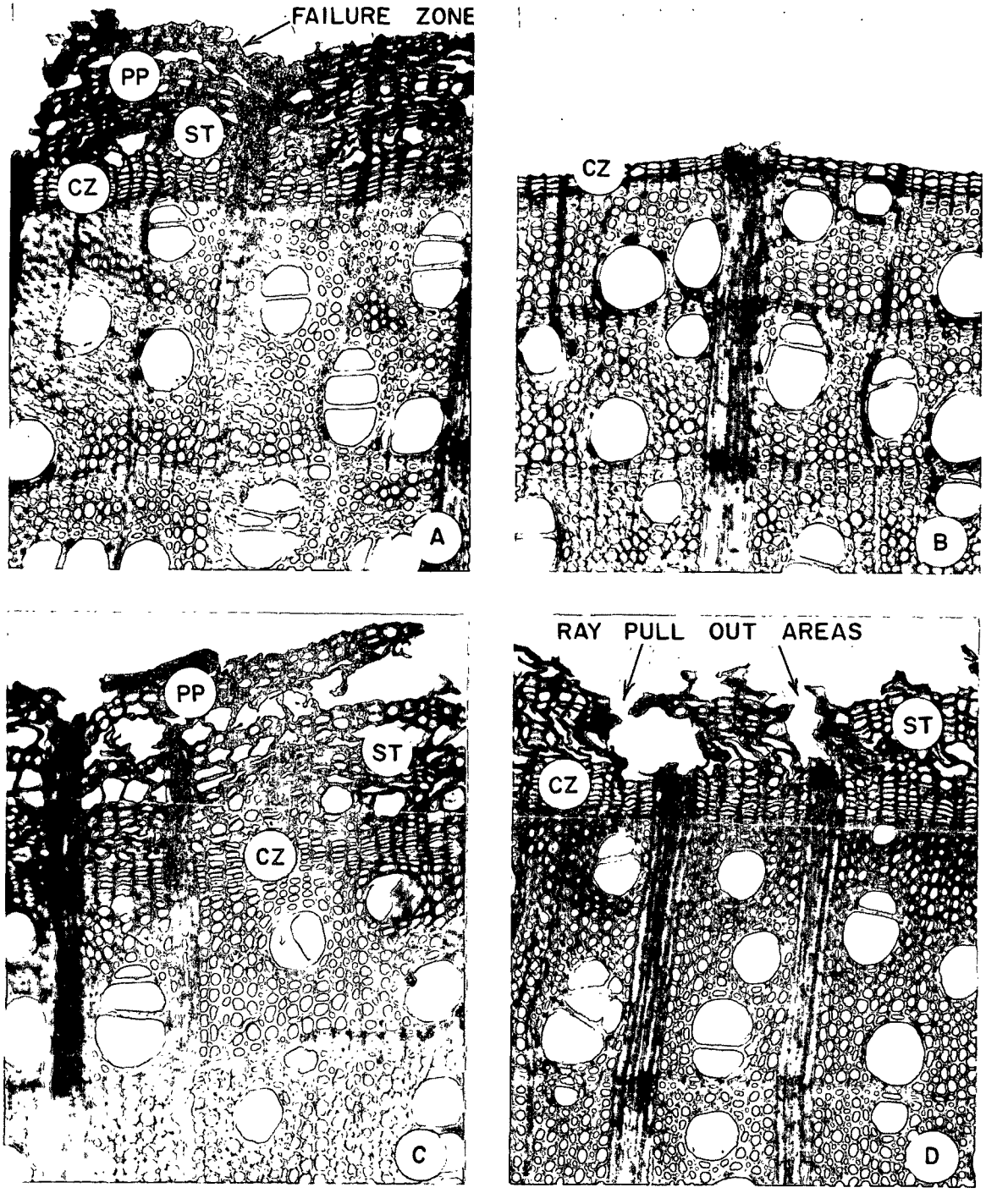

Figure 4. Illustrated are the Seasonal Changes in the Location of the Zone of Failure in Sugar Maple; A — May 18 Collection, Failure in Phloem Parenchyma and Sieve Tube Area (PP-ST) just Outside the Cambium (CZ); B — June 1 Collection, Failure in Cambium Zone (CZ); C — June 29 Collection, Failure in Newly Formed Zone (CZ); D — August 10 Collection, Failure Again in the PP-ST Area with Failure Being Influenced by the "Pulling Out" of Phloem Rays

inside last-formed tangential band of thick-walled, heavily lignified sclerenchyma cells. Estimated adhesion in the cambium zone was in excess of 10.3 kg./cm.^{2}

May 4 — Cambium dormant; cambium zone 6-8 cells in width; failure occurred in the last-formed phloem parenchyma — sieve tube area, a zone which is located between the cambium and the last-formed tangential band of thick-walled, heavily lignified sclerenchyma cells. Starch is abundant in the xylem and phloem ray cells and in the three or four last formed xylem cells. Estimated adhesion in the cambium zone was in excess of 8.7 kg./cm.^{2}

May 18 — Cambium dormant; cambium zone 6-8 cells in width; failure occurred in last-formed phloem parenchyma — sieve tube area (inside the last formed sclerenchyma cells) and partially in the first few undifferentiated cells in the cambium zone, just outside the terminal band of xylem cells (see Fig. 3A and 4A). Adhesion in the cambium zone was in excess of 8.5 kg./cm.^{2}

June 1 — Cambium active; cambium zone 10-12 cells in width, none of the cells in the cambium zone showed lignification; failure zone located in the undifferentiated cells of the cambium zone and 3-4 cells outside the mature xylem cells of last year's growth (see Fig. 3B and 4B). Adhesion in the cambium zone was 5.4 kg./cm.^{2}

June 29 — Cambium active; cambium zone approximately 8 cells in width; 10-12 differentiated and partially lignified xylem cells present; failure occurred primarily between the last-formed sclerenchyma cells and the adjacent tangential bands of phloem parenchyma and crushed sieve

tubes near the cambium (see Fig. 3C and 4C). Estimated adhesion in the cambium zone was in excess of 6.2 kg./cm.^{2}

July 22 — Cambium dormant; cambium zone approximately 6-8 cells in width; all cells in the differentiated xylem area show lignification but lignification is not complete; failure occurred in the same area as June 29 and essentially in the same area as when the trees were dormant in early spring. Zone of thin-walled sieve tubes and adjacent phloem parenchyma very prominent as a zone of weakness. Adhesion in the cambium zone in excess of 7.9 kg./cm.^{2}

August 10 — Cambium dormant; cambium zone is 4-5 cells in width and all cells in this year's growth are fully mature and lignification is complete (Fig. 3D). Failure again occurred in the inner bark, just outside the cambium zone and between the zone of phloem parenchyma cells and the crushed sieve tubes and the bands of last-formed thick-walled sclerenchyma cells (Fig. 4D). Estimated adhesion in the cambium zone was in excess of 10.8 kg./cm.^{2}

August 24 — Cambium dormant; cambium zone is 4-5 cells in width and xylem cells fully mature and lignified. Failure continues to occur primarily in the same zone as reported previously for periods when the cambium is dormant. One unusual feature that was noted in the August 10 and again in the August 24 collection was the tendency for the failure line to travel along the phloem parenchyma and crushed sieve tube area, then down a large ray to the cambium, then across the ray and back out to the parenchyma — sieve tube area. Figure 4D illustrates the tendency of the rays to be pulled out down to the cambium zone.

The ray cells apparently are having an effect on adhesion. Occasionally the xylem half of the tested sample had ray stubs as well as ray pull-out areas. Adhesion in the cambium zone is in excess of 10.2 kg./cm.^{2}

September 14 — Cambium dormant; thickness of cambium zone, lignification and location of zone of failure is essentially the same as in August. The only exception is that the large ray cells apparently have differentiated more in the cambium zone and ray pull-out areas are less prevalent.

September 21 — Cambium dormant and the walls of the cells in the cambium zone have become relatively thick. The contents of the cell lumens are increasing. Other conditions are identical with those reported for September 14. Adhesion in the cambium zone is in excess of 13.2 kg./cm.^{2}

October 5 — Cambium dormant; cambium zone 4-5 cells in width; contents of cells in the cambium zone continue to increase. Starch is abundant in the xylem and phloem ray cells and the last 6-8 rows of summerwood fibers. Failure continues to occur outside the cambium in the inner bark region (phloem parenchyma and sieve tube area) and next to the last formed sclerenchyma (sclereid) cells. The October cross sections also show the existence, for the first time, of phloem fibers located only a few cells outside the cambium. Estimated adhesion in the cambium zone is in excess of 11.6 kg./cm.^{2}

Adhesion varied from 5.4 kg./cm.^{2} on June 1 to 13.2 kg./cm.^{2} on September 21. Based upon wood/bark adhesion measurements and morphological observations,

the peeling season for the maple trees sampled extended from about May 25 to July 17 (wood/bark adhesion less than 7.5 kg./cm.^{2}). The length of the peeling season was somewhat less than expected and believed to be due to the relatively slow growth of the trees involved.

Failure in the test specimens prior to the "peeling season" occurred in the inner bark (secondary phloem) in an area of immature phloem parenchyma and phloem sieve tubes just outside the cambium zone. During the first part of the peeling season the failure was located in the cambium zone and then later moved into a zone of newly formed phloem parenchyma and sieve tubes just outside the cambium. During the dormant period of late summer and early fall the failure zone was again found to be in the phloem parenchyma and sieve tube area just outside the cambium. Phloem maturation is very complicated compared to the simplicity of xylem maturation where all cells produced within a year mature that same year. Only a quarter of the phloem cells produced within one season mature before winter dormancy. The remaining cells initiate or complete maturation the following spring prior to resumption of cambium activity.

Observations made on cross sections during the dormant season indicated the primary zones of weakness are the undifferentiated cells of the cambium zone and the nonlignified, partially matured phloem parenchyma and sieve tubes just outside the cambium. Treatments aimed at reducing adhesion during the dormant seasons will be concentrated on these zones. The observations on the zones of weakness for maple were identical to those obtained for white birch. Also, as with the birch, the presence of well-developed rays apparently influence the adhesion values obtained.

BUR OAK

Anatomical Structure of Wood and Bark

Bur oak is classified as a ring porous hardwood, the springwood vessels forming a conspicuous band 1-3 pores in width. The wood (xylem) of bur oak is made up primarily of fibers, vessels, and ray cells. Longitudinal parenchyma and tracheids are also present with the latter forming most of the conjunctive tissue between the springwood vessels and the rays and making up part of the flame-shaped tracts, along with the summerwood vessels.

The vessels are usually in groups of 5 or more and separated by radially aligned tracts of fibers or by broad rays. The springwood vessels are between 180 and 380 µm. in diameter, while the summerwood vessels average only 35 to 40 µm. in diameter. The summerwood elements appear in radially aligned, flame-shaped tracts, separated by groups of fibers. The fibers are medium thick to thick-walled and average approximately 20 µm. in diameter and 1.4 mm. in length. Gelatinous fibers are fairly common. Bur oak has both broad and narrow rays. The broad rays are unstoried, homogeneous (25+ seriate) and more than 400 µm. in width, while the narrow rays are principally uniseriate and vary from 1 to 20 cells in height.

The inner bark (secondary phloem) of bur oak contains sieve tubes, phloem parenchyma, and sclerenchyma cells. The phloem ray cells are both narrow (mainly uniseriate) and broad (20+ seriate), with the broad ray often having groups of thick-walled sclereids. Figure 5 illustrates the morphology of the described elements. The thin-walled sieve tubes usually occur in groups of 2-5, are polygonal or oval in shape and have an average diameter (uncollapsed) of 30 to 50 µm. The sieve tubes are mostly crushed except for a few bands of cells near the cambium.

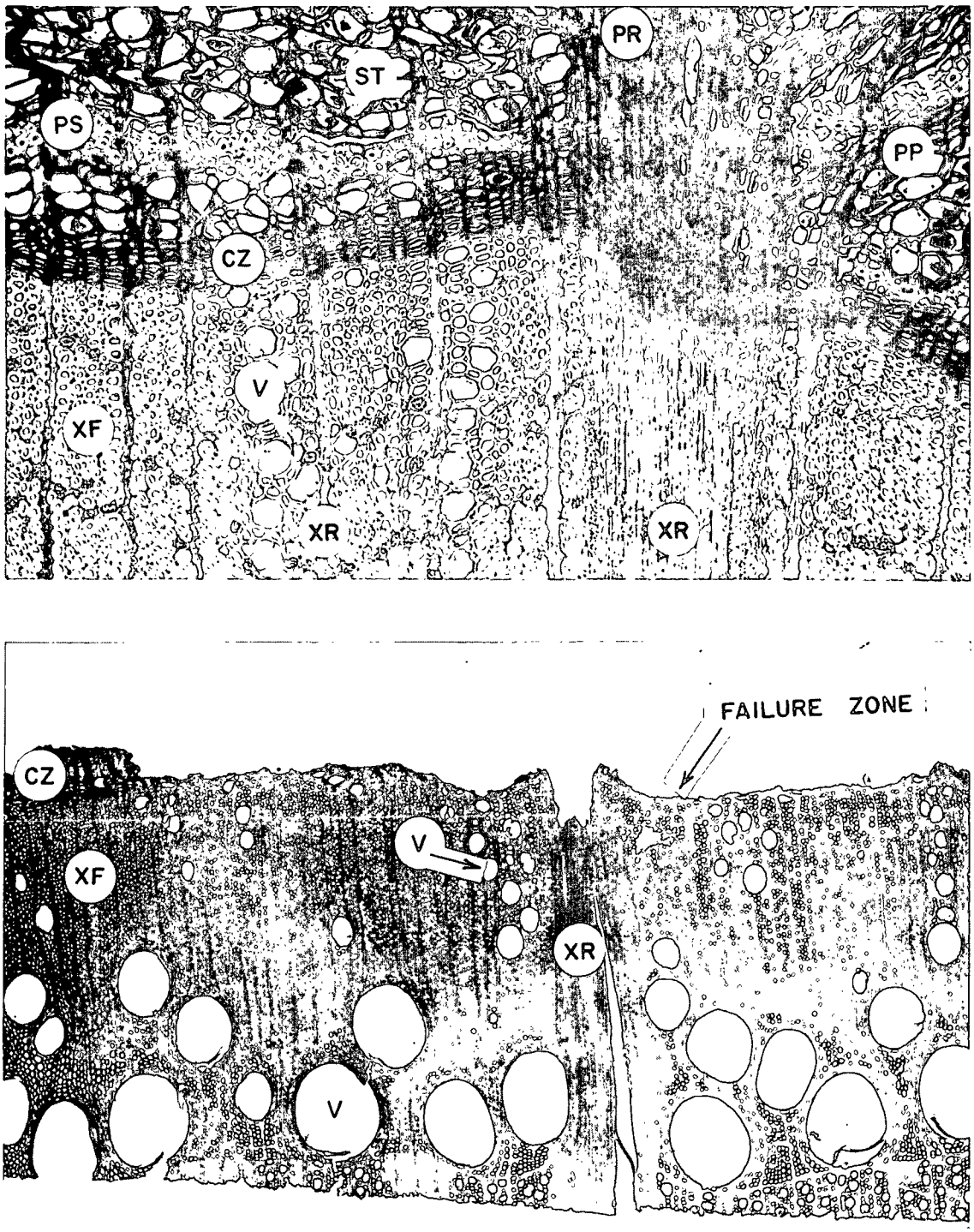

Figure 5. Cross Section of Bur Oak when the Cambium was Dormant (Top, ca. 130X). Illustrated are the Xylem Fibers (XF), Large and Small Xylem Rays (XR), Vessels (V), Sieve Tubes (ST), Phloem Rays (PR), Phloem Parenchyma (PP), Phloem Sclereids (PS), and the Cambium Zone (CZ). The Lower Cross Section (ca. 35X) from a June 29 Collection Illustrates the Size Difference Between the Large Earlywood Vessels (V) and the Relatively Small Summerwood Vessels (V). The Entire 1969 Growth Ring is Visible

The phloem parenchyma cells may be in single layers or in tangential bands of 4 or more cells. They are more or less rectangular in cross sections. Their tangential width is approximately 25 μm. and radial thickness averages approximately 15 μm. The cells are variable in length, ranging from 15-20 μm. to more than 50 μm. Crystalliferous parenchyma bands are common in the secondary phloem of bur oak.

The sclerenchyma cells are principally in the form of fibers which are in discontinuous tangential bands, 2-4 cells in thickness. The fibers average approximately 15 μm. in diameter and approximately 1 mm. in length. They have a narrow lumen and a cell wall thickness of 6-7 μm. The secondary wall of many of these fibers is separated and they appear similar to the gelatinous fibers which are common in the xylem of this wood species. Small groups of thick-walled sclereids are scattered throughout the secondary phloem. These types of cells are also present in the wide rays.

Seasonal Variation in Wood/Bark Adhesion

Seasonal sampling of bur oak wood/bark adhesion was initiated on April 10. Measurements, which were made periodically throughout the growing season, were discontinued after the October 5 samples were taken. Table II summarizes both the morphological observations and the results of the measurements taken using the previously described Instron testing procedure. Figure 6 graphically presents the seasonal variation in bur oak wood/bark adhesion. Figure 7 illustrates the seasonal changes that occurred in the cambium zone, and Fig. 8 demonstrates the accompanying changes that developed in the location of the zone of failure. Described below are brief summaries of the observations made on the seasonal morphological changes associated with changes in wood/bark adhesion.

TABLE II

SUMMARY OF OBSERVATIONS ON SEASONAL VARIATION IN
BUR OAK – APPLETON, WISCONSIN

Date	Adhesion, kg./cm.^{2} Average	Adhesion, kg./cm.^{2} Standard Deviation	Cambium Activity[a]	Width Cambium Zone	New Xylem Cells Total No.	New Xylem Cells No. Non-lignified	No. Immature Phloem Cells	Location of Zone of Failure	Additional Zone of Apparent Weakness
4/6/70	9.5	0.66	D	5-6	0	0	6-8	Inner bark between bands of sclerenchyma fibers and adjacent sieve tubes and parenchyma cells	Cambium zone
5/4/70	7.0	1.01	A	20	0	0	6-8	Cambium zone and adjacent to last year's terminal band of fiber	Phloem parenchyma & sieve tube area of inner bark
5/18/70	7.4	0.71	A	16-18	40	20	6-8	Cambium zone as on May 4	Same as May 4
6/1/70	5.7	0.28	A	16-18	60	20	12-14	Cambium zone	Same as May 4
6/29/70	4.4	0.40	A	14-16	80[b]	20	12-14	Cambium zone and last-formed xylem cells	Same as May 4
7/22/70	4.6	0.50	A	8-10	85[b]	4-6	10-12	Cambium zone and last-formed xylem cells	Same as May 4
8/10/70	5.4	0.51	A	8-10	90[b]	4-6	6-8	Cambium zone and last-formed xylem cells	Same as May 4
8/24/70	10.6	0.45	D	6-8	90[b]	2-3	6-8	Cambium zone and last-formed xylem cells	Same as May 4
9/14/70	8.5	0.47	D	4-5	90[b]	0	6-8	Inner bark in zone of collapsed sieve tubes & parenchyma cells	Cambium zone
10/5/70	10.1	0.52	D	4-5	90[b]	0	6-8	Inner bark as described for Sept. 14	Cambium zone

[a] A = active, D = dormant.
[b] Estimated numbers.

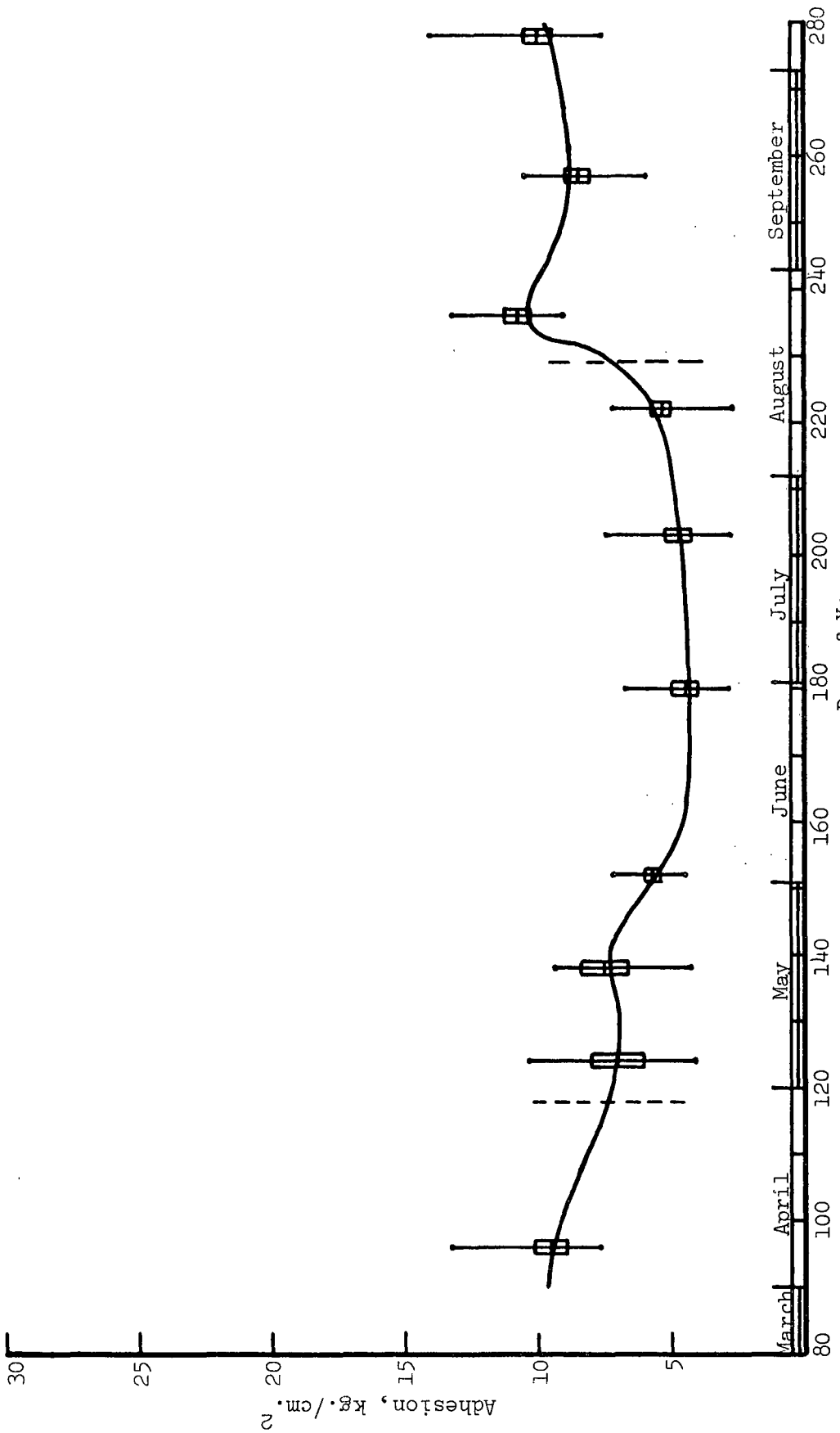

Figure 6. Seasonal Variation in Wood/Bark Adhesion for Bur Oak. Shown for Each Sampling Date is the Range, the Mean and one Standard Deviation each Side of the Mean. The Vertical Dashed Lines Indicate the Estimated Start and end of the Peeling Season

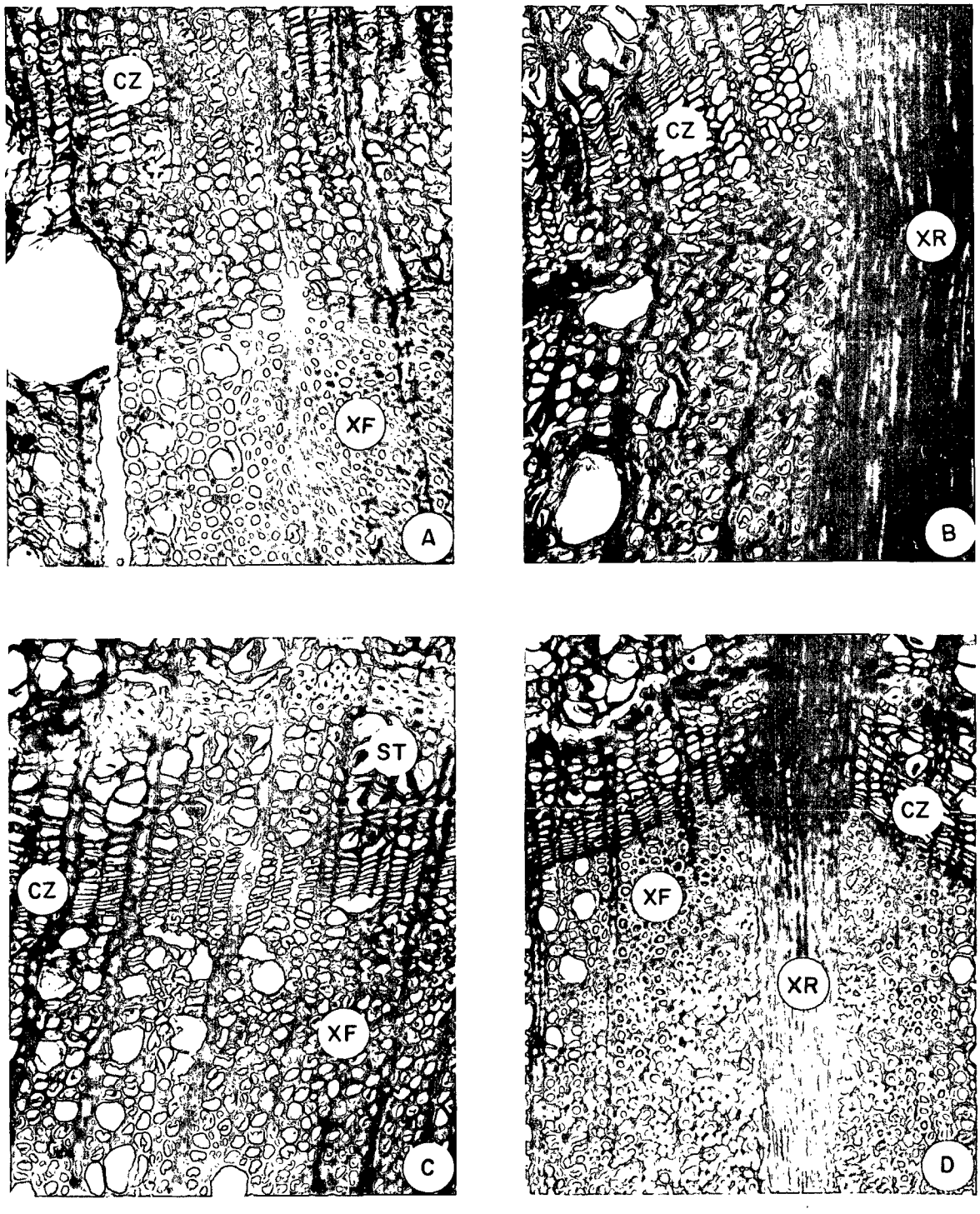

Figure 7. Illustrated, Using Cross Sections of Bur Oak, are the Seasonal Changes that Occurred in the Cambium Zone; A — May 4 Collection, Cambium very Active, Cambium Zone (CZ) 20 Cells in Width; B — June 29 Collection, Cambium Active, First-Formed Xylem Cells Showing Lignification; C — August 10 Collection, Cambium Active, Lignification Evident in all but most Recent Xylem Cells; D — September 14 Collection, Cambium Dormant, all Xylem Fibers (XF) in this Year's Growth Fully Mature and Lignified

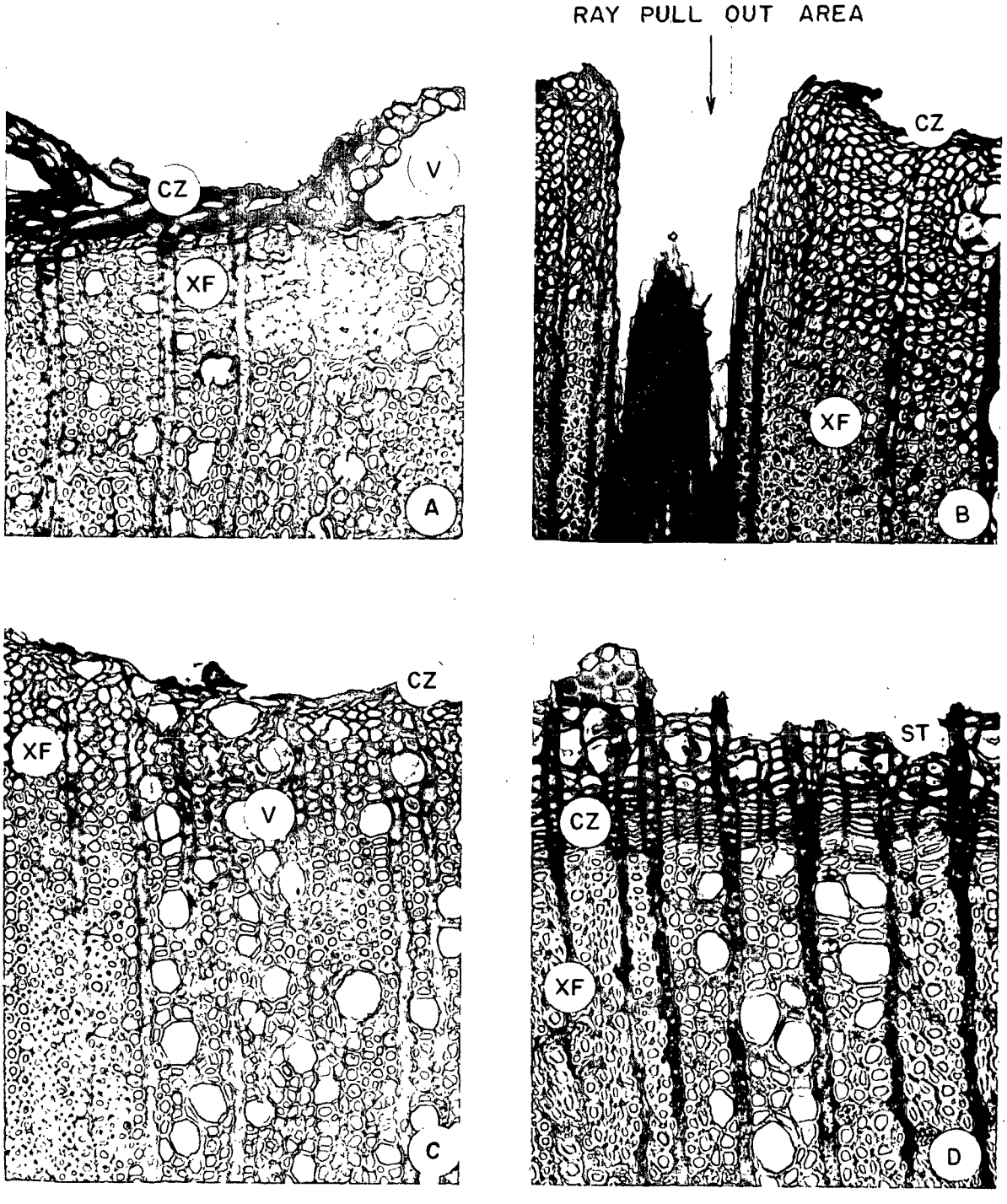

Figure 8. Illustrated are the Seasonal Changes in the Location of the Zone of Failure in Bur Oak. A — May 4 Collection, Failure in the Active Cambium and Xylem Initials just Outside Mature Xylem Fibers (XF) of the Previous Year's Growth; B — June 29 Collection, Failure in Cambium Zone and Nonlignified Xylem Initials, Note Ray "Pull Out Area"; C — August 10 Collection, Failure Occurred in Cambium Zone and Last-Formed Xylem Initials; D — September 14 Collection, Failure Occurred in the Inner Bark in Area of Phloem Parenchyma and Collapsed Sieve Tubes

April 10 — Cambium dormant; cambium zone 5-6 cells in width; starch and other materials evident in the cells of the cambium cells and the xylem ray cells. There were an average of 6-8 thin-walled, uncollapsed sieve tubes between the cambium zone and the strongly lignified thick-walled phloem sclerenchyma cells. Failure occurred in the inner bark (phloem) between the tangential band of sclerenchyma fibers and strands of crystalliferous parenchyma cells and the adjacent sieve tube — parenchyma cell area. Wood/bark adhesion in the cambium zone was in excess of 9.5 kg./cm.^{2}

May 5 — Cambium very active; cambium zone approximately 20 cells in width. Only a very few of the newly formed cells show a differentiation and none show evidence of lignification. Failure occurred between the active cambium and the large differentiated springwood vessels and the nonlignified xylem fibers of the current growth ring (see Fig. 7A and 8A). Also, it was first noted that nonlignified ray cells extended into the previous year's growth ring. Failure of the rays occurred 300-400 μm. into the previous year's growth ring resulting in ray "pull-out area" on the xylem side and "ray stubs" on the phloem side of the failure zone (see Fig. 8B). Wood/bark adhesion in the cambium zone was 7.0 kg./cm.^{2}

May 18 — Cambium active; complete description of the collection was not made. Failure was believed to have occurred in the cambium zone. Wood/bark adhesion in the cambium zone was 7.4 kg./cm.^{2}

June 1 — Cambium very active; cambium zone approximately 16-18 cells in width. This year's growth ring contains approximately 60 differentiated xylem cells. The first formed two-thirds of the newly formed

xylem cells showed lignification. Failure occurred primarily in the cambium cells. Failure of the broad rays extended beyond last year's terminal band of heavily lignified fibers and again created "ray stubs" and "ray pull-out areas" in the failure zone. Wood/bark adhesion in the cambium zone was 5.7 kg./cm.^{2}

June 29 — Cambium active; cambium zone 14-16 cells in width; additional xylem cells being differentiated and lignified. Status of lignification similar to that of June 1. Failure occurred primarily in the cambium zone and in immature, nonlignified xylem initials. Phloem "ray stubs" and xylem "pull-out areas" were prominent (Fig. 8B). Wood/bark adhesion in the cambium zone was 4.4 kg./cm.^{2}

July 22 — Cambium active; cambium zone 8-10 cells in width; lignification evident in all differentiated xylem cells except for the last 4 to 6 rows. Failure occurred in the cambium zone and in the last formed immature xylem initials. Phloem "ray stubs" between 0.5 and 1.0 mm. in length are evident (see Fig. 9) and appear to affect wood/bark adhesion. Wood/bark adhesion in the cambium zone was 4.6 kg./cm.^{2}

August 10 — The state of the cambium, cell lignification and failure of the Instron tested specimens were very similar to that observed for the July sample. Wood/bark adhesion was 5.4 kg./cm.^{2} in the cambium zone.

August 24 — Cambium dormant; cambium zone 6-8 cells in width; all cells in this year's growth increment show lignification; however, lignification is not complete in the last formed 2-3 rows of xylem cells. Failure occurred in the cambium zone immediately adjacent to the

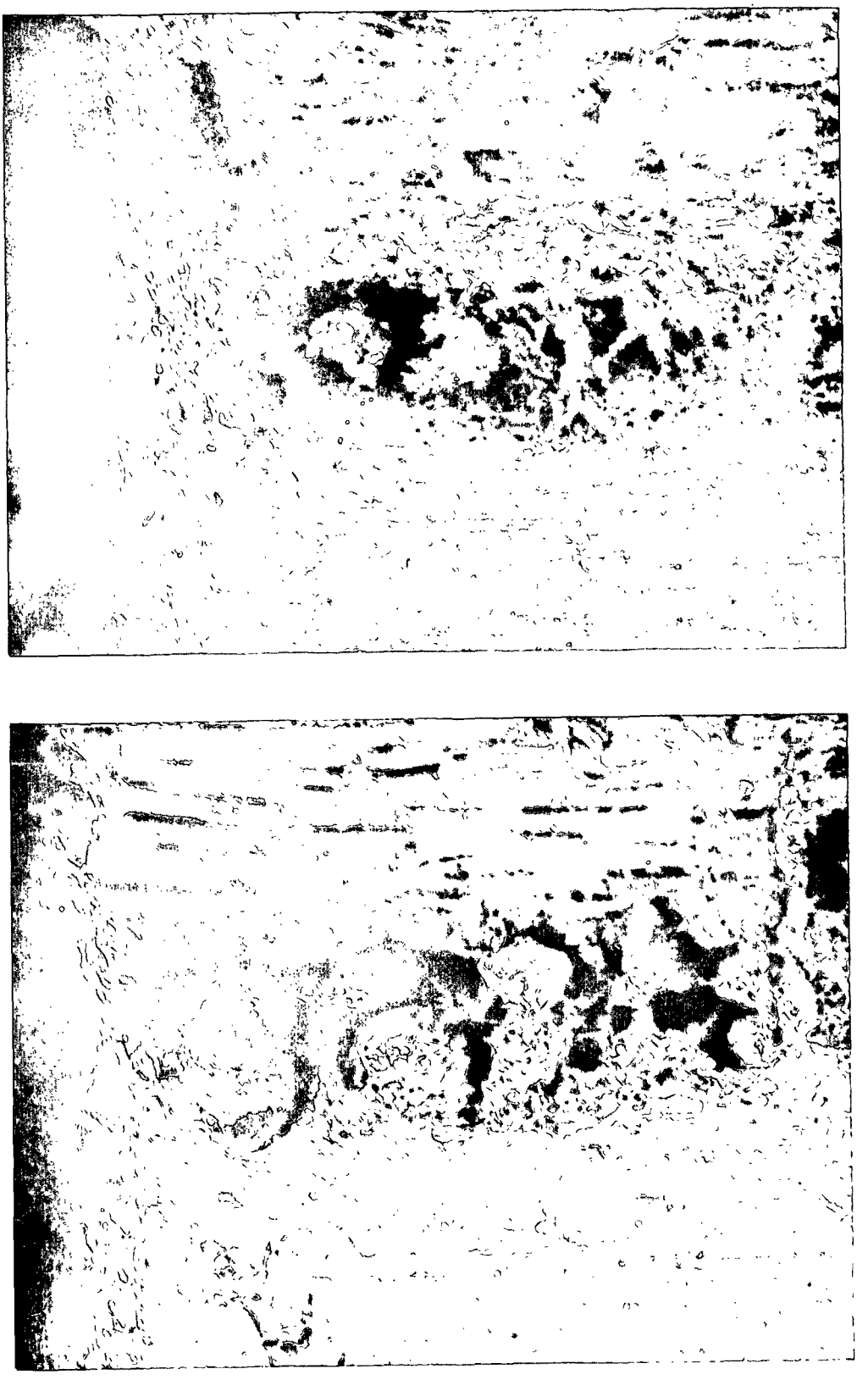

Figure 9. Illustrated are Scanning Electron Micrographs of Bur Oak that Show: (Left) "Ray Stubs" Attached to the Bark and (Right) the Corresponding Wood "Pull-Out Areas." The Photographs are of Samples Collected and Tested in Early May when the Cambium was Active

last-formed xylem cells; break often juts into xylem near region of broad rays. Wood/bark adhesion in cambium zone was 10.4 kg./cm.^{2}

September 14 — Cambium dormant; cambium zone 4-5 cells in width; all xylem cells of this year's growth fully mature and lignification is complete. Failure occurred in the inner bark, primarily in a zone of collapsed phloem sieve tubes and parenchyma cells and inside and adjacent to bands of sclerenchyma fibers (See Fig. 8D). Wood/bark adhesion in the cambium zone was in excess of 8.5 kg./cm.^{2}

October 5 — Cambium dormant; cambium zone 4-5 cells in width; no change in lignification or other developments. Failure occurred in the same inner bark region as described for September. Wood/bark adhesion in the cambium zone was in excess of 10.4 kg./cm.^{2}

The bark peeling season for bur oak was the longest of any of the species tested. Based upon test values, cambium activity, and the width of the cambium zone, it was estimated to extend from April 28 to August 17 (adhesion values less than 7.5 kg./cm.^{2}). Wood/bark adhesion values during the peeling season averaged 5.8 kg./cm.^{2} for oak and was identical to that of maple. The length of the peeling season, as discussed in Progress Report One, can be influenced by such factors as tree vigor, growth rate, site quality, and climatic factors, including temperature and rainfall. In reviewing the site quality information for the bur oak used, it appears that the length of the peeling season for 1971 may be atypical. July and August of 1970 were warm and dry and the oak sample, since it was growing on a moist site and had been thinned recently, apparently benefited from the prevailing climatic conditions. Growth was quite rapid and the period of reduced adhesion was very likely longer than for oak on typical upland sites.

Failure in the test specimens during the "peeling season" started out in the cambium zone in early May and there was little change in the location of these zones of failure until late August. At that time the cambium became dormant the test specimens began to fail in the inner bark, in the immature phloem sieve tube and parenchyma area and inside the older tangential bands of sclerenchyma fibers. Phloem "ray stubs" and xylem "pull-out areas" were characteristic of the failure zone during the period of active cambium growth but were less prevalent during the dormant period. Figure 9 illustrates the abovedescribed phenomenon. The broad rays appeared to "lock" the bark to the wood. However, a between-species comparison of adhesion values failed to demonstrate that the existence of the broad rays increased wood/bark adhesion of oak during the peeling season.

Observations made on cross sections during the dormant season indicate that, as in the case of maple, the primary zones of weakness are the differentiated cells of the cambium zone and the nonlignified, partially-mature phloem parenchyma and sieve tubes just outside the cambium. Treatments aimed at reducing dormant season adhesion will consider these zones of weakness.

Maximum test values for oak suggests that the bark of oak is relatively weak and that during the dormant season the large rays may, in fact, lock the bark on very tightly. The difficulty in peeling oak results during the dormant season when the rays lock on the bark and the weak bark prevents its complete removal via such methods as drum debarking.

BETWEEN SPECIES COMPARISONS

The four tree species (bur oak, quaking aspen, white birch, and sugar maple) upon which measurements have been completed are compared for the reader's convenience in Table III. Included in this table is a summary of information on

length of peeling season, level of adhesion during the peeling season and the dormant season, the principal zones of failure during the dormant season and growing season, and information on wood and bark morphology.

TABLE III

BETWEEN SPECIES COMPARISONS OF ADHESION AND FAILURE ZONES

Species	Peeling Season			Adhesion, kg./cm.^{2}	
	Start	Stop	No. Days	Peeling Season	Dormant Period
White birch	4/20	7/9	80	5.1	12.0
Quaking aspen	4/23	7/9	77	6.4	11.4
Bur oak	4/28	8/17	111	5.8	9.6
Sugar maple	5/25	7/17	53	5.8	10.1

Species	Zone of Failure		Morphology of Inner Bark, Inside Zone of Failure	Number of Large Rays
	Peeling Season	Dormant Season		
White birch	Cambium zone[a]	Immature parenchyma & sieve tubes of inner bark	Phloem fibers, immature & mature sieve tubes and parenchyma. Also scattered sclereids	Many[b]
Quaking aspen	Cambium zone[a]	Same as above	Phloem fibers, immature and mature phloem sieve tube & parenchyma cells	None
Bur oak	Cambium zone[a]	Same as above	Immature and mature phloem parenchyma and crushed sieve tubes	Many
Sugar maple	Cambium zone[a]	Same as above	Immature and mature phloem parenchyma and crushed phloem sieve tubes	Many[b]

[a]Usually in cambium zone or newly-formed xylem cells just inside the cambium zone.
[b]Mostly medium-sized rather than large rays.

Of the several factors compared, the length of peeling season, although important to the pulpwood producer, is the least important to the success of this study and is the value most influenced by environmental factors (climate, soils, etc.) and tree vigor. The length of the peeling season varied from 53 days for sugar maple to 111 days for bur oak. Of importance is the level of adhesion during the peeling season and the location of the zone of failure. Although the species studied varied considerably in wood and bark morphology, the wood/bark adhesion during the peeling season was surprisingly uniform. Adhesion ranged from 5.1 kg./cm.^{2} for white birch to 6.4 kg./cm.^{2} for quaking aspen. Also, the zone of failure during the peeling season was quite consistently located in the cambium zone or the newly formed xylem cells just inside the cambium zone.[3]

During the dormant season the location of the zone of failure for all species tested was quite consistently located in the inner bark. As soon as the cambium became dormant and the xylem cells had matured, failure occurred in the zone of immature and mature parenchyma and sieve tube cells just outside the cambium. The consistent location of the failure zone may have been in part due to the way the samples were prepared for testing although this seems doubtful in view of the visual evidence on cross sections that indicated the presence of large numbers of thin-walled cells in the inner bark region. Dormant season wood/bark adhesion values demonstrated that the weakest zone was not the most recently differentiated xylem cells or the cells of the cambium zone but instead the cells of the inner bark. Based on the dormant season adhesion values, which must be interpreted as a measure of the strength of the inner bark rather than wood/bark adhesion, oak and maple have the weakest inner bark while aspen and birch the

[3]Cambium zone – the true cambium consists of a single layer of dividing cells from which the xylem (wood) and secondary phloem (inner bark) arise. In this study the term "cambium zone" has been used to designate the true cambium plus all undifferentiated xylem and phloem cells immediately adjacent to the cambium.

strongest (Table III). Since many mechanical debarking procedures depend on bark roughness and the strength of the bark, it would appear that low inner bark strength may be a factor in the debarking of oak and perhaps maple.

There existed considerable difference in the size of the rays in the wood and bark of the species tested. The presence of large rays in oak apparently did not increase the wood/bark adhesion during the peeling season and may have, in fact, resulted in reduced adhesion because of the lack of lignification of the ray areas until late summer. During the dormant season, visual evidence suggests that the presence of large rays when mature may, via an interlocking action, cause the bark to be held more tightly. Measurement techniques, because they measure the strength of the inner bark, did not pick up such differences if they did exist.

REDUCING WOOD/BARK ADHESION

INTRODUCTION

A review of the morphological data of the four hardwoods examined reveals a surprising between-species uniformity in the location of the zone of failure during both the dormant season and the season of active cambium growth. This suggests that a method that is successful in reducing wood/bark adhesion with one species very likely will be successful with all four species studied. Also of interest is the difference between species in the level of wood/bark adhesion. During the period of active cambium growth, adhesion values were quite similar. Aspen, which is considered to be easy to peel during the so-called peeling season, actually had the highest wood/bark adhesion values. The dormant season wood/bark adhesion values were less uniform and appeared, because of the nature of the test, to give a measure of the strength of the inner bark.

Examination of failure zones and the morphology of the wood and inner bark regions suggests that during the dormant season the presence of large ray cells may be a factor in locking the bark more tightly to the wood. During periods of active cambium growth, however, ray cells do not appear to be a factor because of the lack of lignification.

Historically, methods of reducing adhesion have received considerable attention. Most investigations have involved treatments of standing trees or pulpwood bolts. Treatments used in this investigation were selected for use on unbarked chipped samples and with the view of being relatively rapid and compatible with existing pulping procedures. At this point, the overall similarity between species in morphology and adhesion has not revealed the hoped-for differences between

species which could be related to reported differences between species in the ease of debarking.

Difficulty has been encountered in establishing a consistent rating as to species considered to be difficult and easy to debark. Also, it has become evident that in many instances species considered difficult to debark are difficult, not because of high wood/bark adhesion, but because of such factors as stringy bark, lack of roughness, or relatively weak bark strength.

PROCEDURES

To facilitate the screening of possible ways to reduce wood/bark adhesion and keep costs at a reasonable level, a procedure using simulated chips was developed. Two sizes of chips were employed. The simulated chips were prepared on a band saw and were tangential sections that contained both wood and bark with the wood/bark interface located approximately equidistant between the wood and bark surfaces. Chips 1-1/2 x 1-1/2 inches and approximately 1/4-inch thick were used in the early trials and the size was later reduced to 1-1/2 x 3/4 x 1/4. Dormant season samples, collected in January, February, and March were treated and tested within 72 hours after collection. Wood/bark adhesion of most of the samples were rated (from 1 to 10) as to ease with which the bark could be removed and only occasionally were tabs prepared and the samples evaluated on the Instron tester. Table IV relates the ranking system to the equivalent Instron test values. Basically, the hope in this series of trials was to reduce wood/bark adhesion to levels equivalent to values obtained during the peeling season (5.1-6.4 kg./cm.^{2}). The treatments applied can be grouped into three categories, namely, thermal, chemical, and mechanical. The discussion that follows describes the use of these three types of treatments to reduce wood/bark adhesion.

TABLE IV

COMPARISON OF ESTIMATED ADHESION VALUES
WITH MEASUREMENTS ON INSTRON TESTER

Adhesion Ranking Values	Instron Test Values, kg./cm.^{2}	Description of Adhesion
1	<2	Falls off to touch
2	2-3	Removed easily by hand
3	3-4	Removed easily by hand
4	4-5	Moderate force required
5	5-6	Considerable force required
6	7-8	Knife required to remove bark — cambium no longer active
7	8-9	
8	10	Moderately difficult to remove with knife
9	10+	Bark removed with considerable difficulty using knife
10	10+	

THERMAL TREATMENTS

Thermal methods were applied with the hope of disrupting the thin-walled cells in the cambium zone and the last-formed phloem cells which are generally higher in moisture content than the surrounding xylem and phloem cells. The treatments used in the attempt to reduce adhesion included hot water, live steam, and an autoclave treatment (temperature plus pressure). Simulated aspen chips[4] were employed and the temperatures were maintained by the use of a thermostat-controlled, electrically heated oil bath for the hot water treatments.

[4]Aspen chips were used in this series of experiments because they were more readily available than the other species being investigated.

Hot Water

The hot water treatment of chips to reduce wood/bark adhesion during the dormant season consisted of treating the simulated aspen chips at either 200, 150, or 100°F. for times of 5, 3, or 1 minute and then estimating the adhesion by determining the amount of pressure or force required to remove the bark at the wood/bark interface. The force required was given a rank from 1 to 10 with 1 indicating bark that fell off upon touching, values from 2 to 5 indicated the bark could be slipped off by hand, values above 5 required a knife to remove, and values of 9 and 10 indicated the bark was removed with a knife with great difficulty.

Live Steam

Live steam, live steam followed by a cold water quench, and hot water followed by a cold water quench, were also tried as possible methods of reducing adhesion. Also, in a later series of treatments, the time of the 200°F. treatment was increased to 10 minutes and this information has been included for comparison purposes.

The results of this series of experiments are summarized in Table V. The hot water treatments, as the data indicate, did reduce wood/bark adhesion and the highest temperature and longer treatment time were the most effective.

The live steam treatment, which was applied by merely playing steam over a small group of chips supported on a screen, was not as effective as the 200°F. treatments. Confining the steam in order to increase the temperature of the chips could be expected to increase the effectiveness of this treatment. Use of a cold water quench, which consisted of dropping the chips into cold water following the treatment, failed to improve the effectiveness of the technique. Based upon the

preliminary results obtained, it appears that a hot-water treatment could be developed that would reduce dormant season adhesion to a level that, with a reasonable amount of mechanical action, the separation of wood and bark could be effected. Either higher temperatures (greater than 200°F.) or treatment times in excess of 10 minutes would be required to drop the estimated wood/bark adhesion below 5.0.

TABLE V

RESULTS OF HOT WATER AND STEAM ATTEMPTS TO REDUCE WOOD/BARK ADHESION

Treatment Description	Temperature, °F.	Time, min.	Estimated Wood/Bark Adhesion[a,b]
Hot water only	200	10	5.8
		5	6.3
		3	6.5
		1	7.0
Hot water only	150	5	8.0
		3	8.0
		1	8.5
Hot water only	100	5	9.0
		3	9.0
		1	9.0
Hot water + 1 minute cold water quench	200	5	6.2
Live steam	?	5	7.8
		3	8.0
		1	7.8
Live steam + quench (1 min.)	?	5	7.5
Control	0	--	9.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b]Values are the average of four determinations.

Autoclave Treatments

Previous experience in the Genetics and Physiology Group with disk samples indicated that a combination of temperature and pressure might provide a promising method of decreasing wood/bark adhesion in chip samples. Table VI summarizes the results of four preliminary trials conducted to examine the feasibility of this approach. Two pressures (7-8 lb./in.^{2} and 15 lb./in.^{2}) and two times at pressure (30 seconds and 3 minutes) were used. A small electrically heated autoclave which could reach pressure and temperature quite rapidly was used. However, since the time to reach pressure and a time to return the sample to zero pressure appeared important, this information was recorded also. As the results indicate, the most effective treatment was the use of 15 lb. pressure and a time at pressure of 3 min. This treatment attained a maximum temperature of 252°F. and had a total treatment time of 12-3/4 min. The 15 lb.-30 sec. treatment was also quite effective and resulted in a total treatment time of approximately 9 min. Only a minor amount of mechanical action would be required to effect separation at the wood/bark interface for chips treated in this manner. Examination of the failure surfaces indicated failure was occurring in the cambium zone.

TABLE VI

AUTOCLAVE TREATMENTS TO REDUCE WOOD/BARK ADHESION ASPEN

Trial No.	Pressure, lb./sq.in.	Temp., °F.	At Pressure	To Pressure	To Zero Pressure	Total	Estimated Adhesion[a,b]
1	7-8	230-234	3'0"	3'25"	2'5"	8'30"	5.5
2	7-8	228-230	0'30"	1'15"	2'25"	4'10"	8.0
3	15	252	3'0"	6'20"	3'25"	12'45"	2.0
4	15	251	0'30"	5'7"	3'21"	8'58"	3.0
5[c]	0	70	--	--	--	--	9.0

[a]Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — requires knife to remove; 10 — bark comes off with great difficulty and tends to sliver.
[b]Values are the average of four determinations.
[c]Control samples tested at room temperature.

CHEMICAL METHODS

Dilute Acids

Interest at The Institute of Paper Chemistry in loosening the bark of pulpwood species is not new. In 1962 and 1963, Dr. R. E. Kremers, as part of his investigations in "cambial chemistry" of pulpwood species[5], noted that during the dormant season cells in the cambium zone were thin walled, low in lignin, and relatively rich in pectic substances. Also, knowing from his work in the field of food chemistry that fruit pectin could be extracted using dilute acids, Dr. Kremers established a series of treatments aimed at reducing wood/bark adhesion. Dormant season disk samples were used for most of the trials and the time it took for the bark to loosen was the basis used for evaluating the results.

The results of these investigations were published by Haas and Kremers (2) and a copy of their paper is included in the Appendix of this report for your convenience. Briefly, this study indicated that sulfurous acid (2.0N) and 0.5-2.0N hydrochloric, sulfuric, sulfamic, and oxalic acids were effective in reducing adhesion to levels equivalent to those prevalent during the growing season. The reaction was sensitive to concentration, time, and temperature; penetration of the acid, too, along the cambium was an important factor. Comparable concentrations of sodium hydroxide or salts did not have the same effect. Successful tests were made with black ash, quaking aspen, white birch, American elm, jack pine, silver maple, sugar maple, and black spruce. The principal disadvantages of the approach is the relatively long treatment time required, the discoloration of the wood that resulted for certain species, and the lack of effectiveness of the treatment on dried samples. In view of the magnitude of the study undertaken

[5]Institute of Paper Chemistry Research Project 1702.

under IPC research Project 1702, no further preliminary work with dilute acids has been undertaken. It appears, however, that by working on chip size, temperature, and ways of increasing acid penetration, a satisfactory method employing dilute acids could be developed.

Waste Pulping Liquors

The principal short-term chemical treatment tried in an effort to reduce wood/bark adhesion consisted of trying a series of waste pulping liquors at temperatures of 100, 150, and 200°F. and at treatment times of 5, 3, and 1 minutes. Table VII briefly describes the waste liquors involved. This somewhat empirical preliminary trial was aimed at investigating the possibility that the chemicals present in the waste liquors might prove to be more effective than water alone in reducing wood/bark adhesion. Wood/bark adhesion was estimated using the previously described ranking system and simulated aspen chips were used in all of the trials.

TABLE VII

DESCRIPTION OF LIQUORS

Type	pH	Strength	Description
NSSC	6.4	Digester strength	Sodium-base liquor from Green Bay Packaging
Sulfite	2.7	Digester strength	Calcium-base liquor from Consolidated Papers, Inc.
Kraft	13.0	Weak black liquor	Standard kraft from Thilmany Pulp & Paper Company
Modified soda	12.6	Recausticized	Black liquor from IPC experimental cook with an original pH of 8.0

Table VIII summarizes results of this series of preliminary chemical treatments. Results indicate that none of the waste liquor treatments were more effective than water alone. Examination of the chips indicated that very little

penetration was being obtained along the cambium zone. Discussions with individuals in pulping research indicate considerably longer treatment times would be required before any appreciable cambium zone penetration would result. In view of the results obtained with dilute acids, hot water, and the autoclave treatments, time and temperature appear to be the factors that can most easily be manipulated to effect disruption and weakening of the adhesion in the cambium zone. Based upon the Instron measurement taken during the dormant period and during the season of cambium activity, it appears adhesion values must be reduced to at least 5-6 (5-7 kg./cm.^{2}) in order that mechanical treatment, such as crushing using pressure rolls, will result in effective separation.

TABLE VIII

RESULTS OF WOOD/BARK ADHESION TEST
WASTE PULPING LIQUOR — ASPEN

Temperature Description	Time, min.	Waste Pulping Liquor — Estimated Adhesion[a,b]				
		NSSC	Sulfite	Kraft	Modified Soda	Water
200°F.	5	6.5	6.8	7.0	6.8	6.5
	3	8.0	7.8	7.8	7.8	6.5
	1	8.2	8.2	7.8	8.8	7.0
150°F.	5	8.5	8.2	8.0	8.5	8.0
	3	8.8	8.5	9.0	8.5	8.0
	1	8.8	8.8	8.5	8.8	8.3
100°F.	5	8.5	9.0	8.5	8.8	9.0
	3	8.5	9.0	8.8	8.8	9.0
	1	9.0	9.0	9.0	8.8	9.0
Control	--	9.0	9.0	9.0	8.8	9.0

[a] Ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

[b] Values are the average of four determinations.

MECHANICAL METHODS

Commonly employed debarking methods use various types of mechanical action to remove bark from logs and pulpwood bolts. Based upon morphological examination of the four hardwoods under investigation (aspen, oak, maple, and birch), similarities in the cambium zone and inner bark region indicate that one of several conventional debarking devices should be equally effective in debarking the four abovelisted species. Such would be the case if the debarking causes a separation in either of the abovedescribed zones. Existing devices are, however, not equally effective on the various hardwood species. Discussions with individuals involved with mechanical debarking indicate that growing season debarking difficulties are apparently due, not to wood/bark adhesion but to the properties of the bark. Dormant season debarking problems, as might be expected, result from a combination of high wood/bark adhesion and some unusual physical property of the bark. Basswood and American elm, for example, are described as being difficult to debark but it appears the problem is not wood/bark adhesion as much as it is the stringy nature of the bark that wraps around the cutting tools and tension arms and after a short period of time renders certain types of barkers inoperative. White birch is described as being a problem species in debarking because the cutter head type of debarkers tend, at times, to run not at the cambium but between the outer papery bark and the reddish-colored inner bark.

Chipping prior to debarking is one approach that would eliminate the types of debarking problems described above. Use of specially modified chippers aimed at producing not only suitable chips but designed to provide the necessary additional mechanical action to cause wood/bark separation in the cambium zone and/or inner bark region seem to have considerable promise. With the above considerations in mind, observations were made on the Institute's 41-inch, 4-knife

Carthage chipper with regard to the kind of separation being obtained under standard operating conditions[6]. These observations were recorded as follows:

Chipper Trial I — A quaking aspen sample collected in November was chipped in a nonfrozen condition. Separation at the wood/bark interface was good. Failure occurred mainly in the cambium zone. Less than 10% of the bark chips had wood attached. Hand separation of the bark and wood resulted in the samples separating at the cambium zone.

Chipper Trial II — Several white birch pulpwood bolts (5-7 inches in diameter) were collected in November and chipped 3-4 days after collection. Separation of bark and wood was quite good although not as satisfactory as with aspen. Approximately 25% of the bark chips had wood attached and when wood and bark was separated by hand, failure usually occurred in the sieve tube area just outside the cambium, leaving a reddish film 2 or 3 cells thick. Some separation occurred in the cambium zone. Separation by the chipper action occurred in the same zone as when separated by hand.

Chipper Trial III — November-collected maple pulpwood bolts were chipped 2-3 days after collection. Separation of wood and bark by the action of the chipper was good with less than 10% of the bark chips having wood attached. When hand separation was required and when the chipper action separation was examined, it was found that a thin layer of inner bark remained on most of the wood samples. In

[6]The chipper runs described were conducted as part of Project 2977. Wood and bark chips were being prepared for use in the work on the characterization of the flotation behavior of wood and bark of several tree species.

appearance the inner bark left on the wood chips was much like that of birch and appeared to be only a few cells thick.

Chipper Trial IV — Bur oak pulpwood bolts collected in mid-November were held and chipped in December. The bolts were not frozen when chipped. Separation of wood and bark via the chipper action was good. An estimated 10% or less of the bark chips had wood attached. The inner bark of the wood when freshly peeled is difficult to tell from the wood but discolors within 24 hours. A thin layer of inner bark was left on most wood chips indicating separation was occurring in the sieve tube area just outside the cambium.

These preliminary observations on the action of a conventional chipper suggests that for the species examined the mechanical action of the chipper quite successfully causes failure, during the dormant season, in those zones of weakness (cambium zone and sieve tube and phloem parenchyma of the inner bark) that became apparent when the morphological observations were summarized (Table III). Experimentation with chipper knife design, chipping angle, and ways of forcing the chips at high velocities against ridged baffles can be expected to further improve the effectiveness of this approach to wood/bark separation.

COMPARISON OF METHODS

A review of possible methods of reducing wood/bark adhesion revealed that, in addition to the thermal, chemical, and mechanical methods that have been examined in a very preliminary way, there seemed to be a possibility of disrupting the cells of the cambium zone and reducing wood/bark adhesion using electrical and/or ultrasonic techniques.

Of the methods examined, the thermal and mechanical seemed to have the most promise. In addition to the previously described trials that were run using simulated aspen chips, the most effective autoclave treatment (15 lb., 250°F., and 3 min.) was applied to simulated chip samples of bur oak, white birch, sugar maple, and quaking aspen. Table IX summarizes the results. The treatment was equally effective on all four species and adhesion was reduced to a level similar to that encountered during the sap peeling season. Some of the treated chips were stored moist for 24 hours after treatment and the adhesion after storage for aspen, birch, and maple was comparable to the values obtained from samples tested immediately after treatment. Storing treated oak chips resulted in higher wood/bark adhesion. No work has been conducted with regard to the effect of drying the treated samples prior to separating wood and bark.

Samples of the above autoclave-treated chips (15 lb., 3 min. treatment) were stained, embedded, and sectioned. Examination of the failure zone revealed that quite consistently failure occurred in the cambium zone with 3 to 4 layers of the cambium zone cells normally remaining attached to the wood. Staining, embedding, and sectioning was also used in examining the failure zone for several treatments made on aspen simulated chips in which the adhesion values were reduced to an estimated 5-6 (5 to 8 kg./cm.^{2}) but were not slipping readily. In these instances failure did not occur in the cambium zone but developed in the sieve tube and phloem parenchyma cells of the inner bark region. For the most part, the failure zone was located approximately 0.2 mm. outside the cambium zone with the result that some thin-walled phloem cells were left on the wood chips. The last-formed inner bark region of the species studied do not contain any appreciable numbers of objectionable thick-walled sclereid cells. Consistent separation in the inner bark region near the cambium zone apparently would constitute a

satisfactory solution to the problem. Additional studies are planned during the fall and winter of 1971-1972 on ways of reducing wood/bark adhesion. All eight species under investigation will be considered and additional treatment techniques will be employed.

TABLE IX

RESULTS OF AUTOCLAVE TREATMENT TO REDUCE WOOD/BARK ADHESION ON DORMANT SEASON SIMULATED CHIPS OF BIRCH, MAPLE, OAK AND ASPEN

Species	Pressure, lb.	Temperature at Pressure, °F.	Time at Pressure, min.	Total Treatment Time, min.	Storage Time before Testing, hr.[a]	Estimated Adhesion[b]
White	15	251	3	11-3/4	0	4.2
birch	15	251	3	11-3/4	24	4.5
	Control	--	--	--	0	9.0
Sugar	15	251	3	11-3/4	0	4.5
maple	15	251	3	11-3/4	24	4.5
	Control	--	--	--	0	10.0
Bur	15	251	3	13-3/4	0	4.0
oak	15	251	3	13-3/4	24	6.0
	Control	--	--	--	--	9.0
Quaking	15	250	3	10	0	4.2
aspen	15	250	3	10	24	4.2
	Control	--	--	--	--	8.0

[a] Simulated chips were stored moist after treatment.

[b] Adhesion ranking — 1 — bark falls off to touch; 5 or below — can be removed by hand pressure, above 5 — require knife to remove; 10 — bark comes off with great difficulty and tends to sliver.

PLANS

Measurement of the seasonal variation in wood/bark adhesion for slash pine, a southern source of cottonwood, shagbark hickory, and white spruce is well on the way and will be completed by November. Morphological observations will be made on samples collected during the early spring dormant season, spring peeling season, summer peeling season, and the late fall dormant season. Adhesion measurements will be related to the observed morphological changes. Dormant, winter condition samples will be collected for all species under investigation. Additional studies will be initiated into ways of reducing wood/bark adhesion on chip samples. In addition to trying several of the most promising preliminary treatments on the four new tree species, methods of improving chemical penetration, increasing the mechanical "chipper action," and improving the effectiveness of the thermal techniques will be investigated. The possibility of using ultrasonic treatments as a method of reducing adhesion will also be reviewed and preliminary trials will be undertaken should the procedure appear feasible.

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LITERATURE CITED

1. Chang, Y.-P. Anatomy of common North American pulpwood barks. TAPPI Monograph Series No. 14, 1954. 249 p.

2. Haas, B. R., and Kremers, R. E. Loosening bark from pulpwood with dilute acids. Tappi 47, no. 11:726-8(1964).

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BARBARA R. HAAS
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Loosening Bark from Pulpwood with Dilute Acids

After materials which had been taken from dormant trees had been treated with dilute acid, the bark peeled as readily as during the growing season. Sulfurous acid (2.0N) and 0.5 to 2.0N hydrochloric, sulfuric, sulfamic, and oxalic acids were effective. The reaction was sensitive to concentration, time, and temperature; penetration of the acid to or along the cambium was an important factor. Comparable concentrations of sodium hydroxide or of salts did not have the same effect. Successful tests were made with black ash, quaking aspen, white birch, American elm, jack pine, silver maple, sugar maple, and black spruce. But dried-out material did not react with acids in the same way as dormant (green) materials. Aspen chips made from a log which had been debarked with sulfurous acid yielded a kraft pulp and handsheets comparable to usual aspen kraft pulp and paper. However, data for an economic appraisal are not yet available.

THE effective, economical removal of bark from pulpwood continues to be an important problem. The endeavor, on the one hand, to prolong the period of easy debarking by killing trees with chemicals during the growing season has not been successful. On the other hand, the use of the perfected portable debarking machines tends to be wasteful.

The ease of separating bark and wood during the growing season depends on the frequent cell divisions in the cambium and the consequent presence of very fragile, watery cells. Conversely, when the cambium becomes dormant and all of the new wood cells have matured, i.e., become hard and drier, the bark and wood adhere firmly. In the next season the return to the sap-peeling condition follows the resumption of cambial activity in the spring which is ascribed to the action of hormones (*1*). Moreover, dormancy can be broken in other plants by appropriate chemical treatments (*2*). Accordingly, it seemed logical that the proper application of hormones would activate dormant cambium and that thereby the conditions which favor bark peeling could be induced artificially. Our experiments did not achieve a controllable result, but did lead to a simple chemical procedure which has proved to be very effective in the laboratory.

It is well established that the pectic materials are prominent in the early stages of the transformation of cambial cells into wood, i.e., before the secondary, thicker cell wall has begun to develop (*3*). Inasmuch as the cells in the dormant cambium are thin walled and unlignified, it is assumed that the cambium also is relatively rich in pectic materials. The observation that the plant hormone indole-acetic acid may promote cell growth by initiating the synthesis of pectin fits with the facts of cell-wall composition (*4*). It is also known that fruit pectins can be extracted with dilute acids. Hence, it was thought that acid might effect a loosening of the bark by acting on the pectic materials in the dormant cambial cells. Experiments based on this premise led to the following results.

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MATERIALS

Materials for these experiments were taken from mid-October until about mid-February, that is, well after the onset of dormancy and before the onset of growth in the spring. In most instances, saplings of 1–2 in. diam., or branches of about the same size, were employed. In order to make certain that the results were not limited to materials of this size and age, trunk sections up to 6-in. diam. were also treated.

METHODS

The stems or branches were cut into 1-in. lengths and the rough edges trimmed; depending on their size, five to seven sections were placed in a flat bottom glass dish, covered with a known volume of the treating acid or other chemical, placed in a vacuum distilling pot, and subjected for 5 min to reduced pressure created by a water pump. The release of the vacuum forced some of the solution into the porous elements of the bark and wood. With sulfurous acid and bisulfite solutions the volatility of the sulfur dioxide required evacuating the wood in a dry chamber, introducing the solution into the vacuum, and allowing the sulfur dioxide which had flashed out to be reabsorbed. Alternatively, dry sulfur dioxide could be introduced first and water added subsequently.

The strength of the treating acids or alkali ranged from 0.1 to 2*N*; comparable concentrations of salts were likewise tested. The reaction times ranged from about 4 hr to 5 days. When materials in 1 or 2*N* acid were set aside overnight, no effort was made to observe the incidence of peeling in the interval between about 4 to 18 hr. Similarly, materials in 0.5 or 0.1*N* acid were observed only at 24 hr intervals after the first day. Most of the experiments were at room temperature; some were at roughly 70°C in an electrically heated oven; a few trials were made at about 100°C.

The effects of the treatments were observed by making a longitudinal incision in the bark and peeling the bark. The criterion for a successful treatment was that the bark separated easily, completely, and cleanly from the wood at the zone of cambial contact; with such a result the appearance of the freshly exposed surfaces was very similar to that of wood and bark separated normally during the growing season.

Table I. Acid Debarking: Quaking Aspen

		Time required to loosen bark	
Acid	*Normality*	*Room temp., hr*	*70° ± 5°*
HCl	2.0	4	4
	1.0	18	4
	0.5	18	4
	0.1	44 (?)	26[b]
$H(NH_2)SO_3$	2.0	18	4
	1.0	18	4
	0.5	18	4
	0.1	(?)	(?)
H_2SO_3	2.0	24	No test
	0.5	96	No test
	0.1	(?)	No test
H_2SO_4	2.0	18	4
	1.0	18	4
	0.5	44	22[c]
	0.1	68 (?)	(?)
$H_2C_2O_4$	2.0	18	4
	1.0	18	4
	0.5	44	22
	0.1	(?)	(?)
Chips	2.0	4	No test
H_2O Control		>92	No test

? = Uncertain result.
[a] Approx. 1-in. stem cylinders.
[b] 22-hr room temp. after 4 hr at 70°.
[c] 18-hr room temp. after 4 hr at 70°.

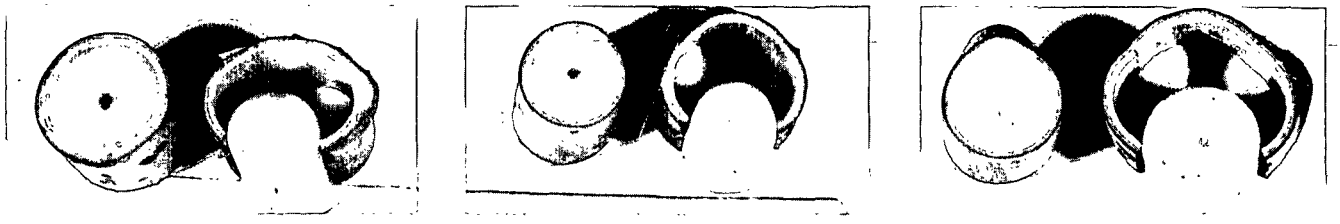

Fig. 1. Quaking aspen, 1N acid-treated cylinders before incision and after "slipping" the bark: (left) hydrochloric; (center) sulfuric; (right) oxalic acid

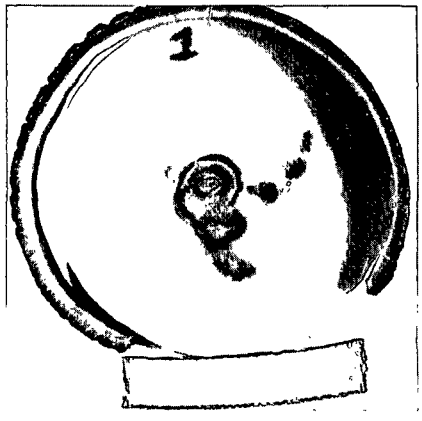

Fig. 2. Quaking aspen, disk from base of tree treated with 1N hydrochloric acid, after "slipping" the bark

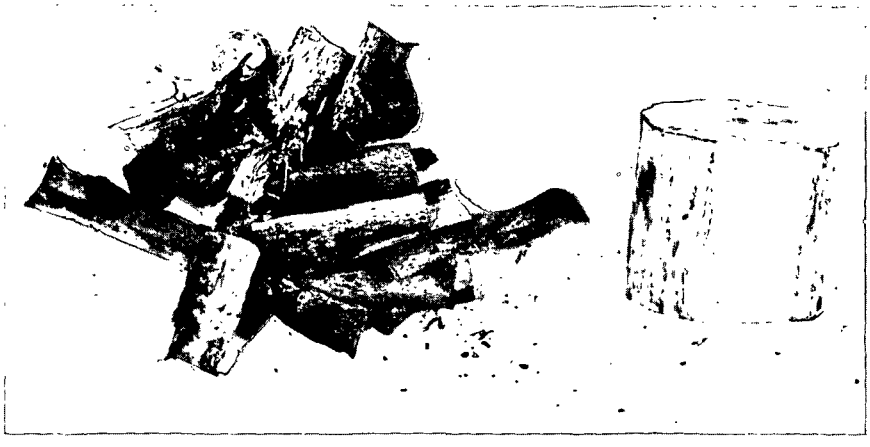

Fig. 3. Quaking aspen, control (in water). Note that inner bark adheres firmly to wood surface, discolors on exposure to air

RESULTS

Typical data for quaking aspen have been assembled in Table I. They show that the reaction is sensitive to concentration, time, and temperature. It is apparent that for hydrochloric, sulfuric, sulfamic, and oxalic acids, the 0.1*N* concentration is at or below the limit of effectiveness at room temperature. The 0.5*N* solutions are more effective than the 0.1*N*, and 1 and 2*N* strengths loosen the bark within 18 hr (Figs. 1–3). Heating the materials for 4 hr at about 70°C loosens the bark with the stronger solutions, and sometimes suffices for the 0.5*N* strengths; 0.1*N* strength is still borderline. Controls in water at room temperature never exhibited loosening of the bark unless held so long that decomposition by microorganisms was extensive.

In the tests of sulfurous acid with the 1 in. cylindrical material only the 2*N* acid was effective at room temperature. But it is noteworthy that with small "chips" the loosening occurred much more rapidly. The chips were flat pieces of bark and sapwood, roughly 1 × 3/4 × 1/4 in. prepared as follows: disks 1 in. thick were cut from trunk sections; outer bark and stone-cell layers were trimmed off until the inner bark (phloem) was exposed; then a hammer and chisel were used to cut longitudinally through the sapwood just within the last growth ring, breaking off the chips. The inference from this experiment is that the penetration of the acid to or along the zone of cambial contact between bark and wood is an important factor in causing the desired effect. Two other observations support this inference: (a) with good impregnation it was not necessary to keep cylinders in contact with the unabsorbed excess of acid; (b) a color change in the bark proceeded from the cut ends toward the midsection of the cylinder and apparently paralleled the loosening effect of the acid.

As indicated in Table II, equally good results were obtained with the other pulpwood species tested: black ash, white birch, elm, jack pine, sugar maple, silver maple, and black spruce. In the case of the elm and silver maple, the materials were taken in mid-October before frost; accordingly, the success of the treatment does not depend upon the tree having first been exposed to winter temperature.

On the other hand, the acid treatment is not effective with dried out, dead materials (Figs. 4 and 5). In this case the acid tends to disintegrate the bark; the cambial layer and some of the inner

Table II. Other Tree Species Tested[a]

	24-hr acid treatment								
	HCl			H_2SO_4			$H_2C_2O_4$		
Species	2*N*	1*N*	0.5*N*	2*N*	1*N*	0.5*N*	2*N*	1*N*	0.5*N*
Black ash	+	+	+	+	+	+	+	+	+
White birch	+	+	+	+	+	+	+	?	+
American elm	+	+	+	...					
Jack pine	...	+	...						
Silver maple	+	+	?					+	
Sugar maple	+	+	+	+	+	+	+	?	+
Black spruce	+	+	+	+	+	+	+	+	+
Quaking aspen									
Branch	+								
Trunk, base[b]	+								
Middle[b]	+								
Top[b]	+								

[a] 1-in. cylinders from saplings, except as noted.
[b] 1-in. disks.
+ = satisfactory peeling.
? = uncertain result.
... = no test.

Fig. 4. Quaking aspen, felled Jan. 20, 1962, treated Dec. 10, 1962; note that bark has turned black during seasoning, will not peel, and that a portion of inner bark still adheres to wood surface

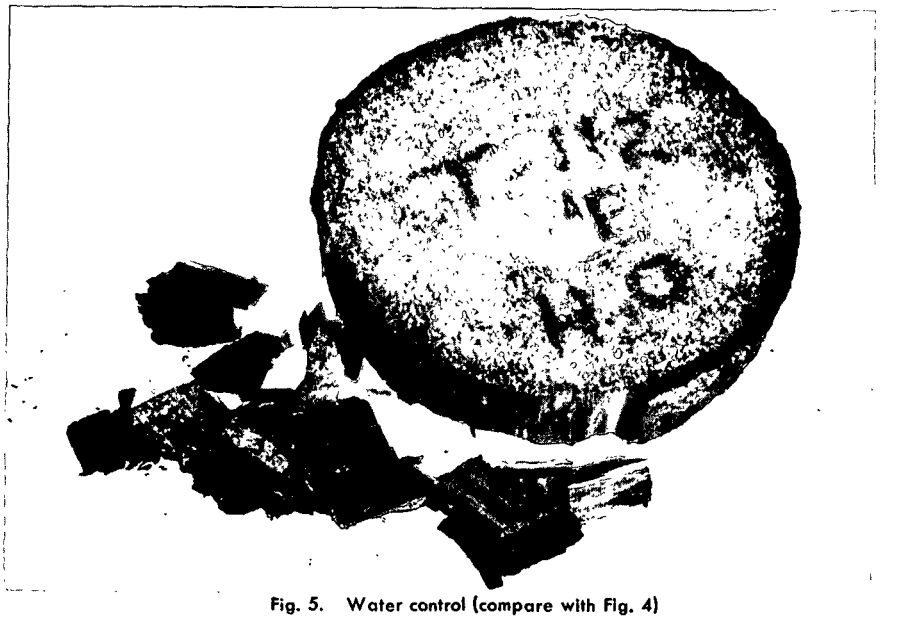

Fig. 5. Water control (compare with Fig. 4)

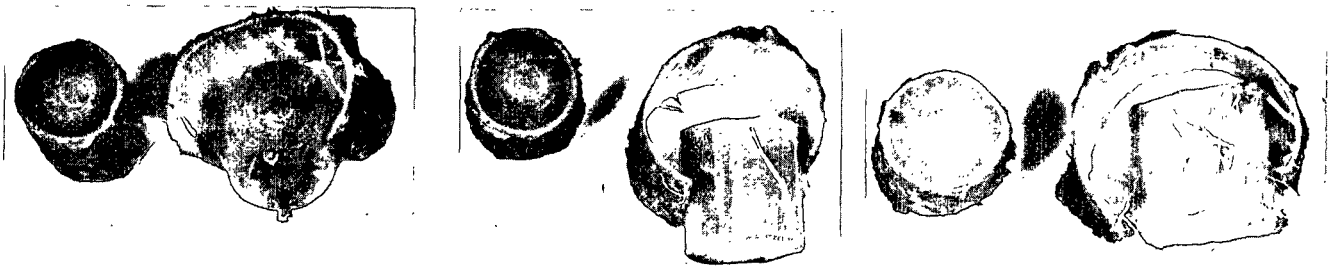

Fig. 6. Black spruce cylinders treated with 1N acid, before and after "slipping" the bark; note dark (purple) color especially in left and center: (lef hydrochloric; (center) sulfuric; (right) oxalic acids

bark tend to stick to the wood; the separation is not clean.

Solutions of sodium hydroxide, sodium bisulfite, sodium sulfite, sodium chloride, potassium chloride, ammonium sulfate, and ammonium oxalate were not effective. The ammonium salts discolored the wood rapidly. The sodium hydroxide, and to a lesser extent the salts, made the bark soft and mushy. This effect was apparently a general degradation and did not in any way simulate natural bark peeling. In this context alkali made debarking a messier, more difficult operation. However, the distinction between the effects of the acids and the other reagents, including water, was greatly lessened when the tests were made at 90–100°C.

A noteworthy incidental result was the red-purple color developed by jack pine, black spruce (Fig. 6), white birch, and sugar maple treated with 2 and 1*N* hydrochloric or sulfuric acids. Black ash turned green; quaking aspen did not discolor, except to turn yellow on contact with sulfamic acid. Oxalic acid produced color only with the spruce. The red-purple color recalls the Isenberg-Buchanan reaction (*5*).

Experiments with longer log sections to obtain data which could be related to commercial practice have been planned, but only a few have been carried through. With a stationary digester that holds a 6 × 26-in. log section, it has been demonstrated that 2*N* sulfuric acid achieved the same result in 24 hours as laboratory tests. Trials with 26-in. sections and 2*N* H_2SO_3 encountered some obstacles: (a) the prevailing, very low, winter temperatures at which the logs were stored apparently prolonged the reaction time more than was anticipated, and (b) the necessary penetration was not always attained. But a later test with 2, 4, 8, and 12-in. sections from a log stored at about +5°C gave satisfactory peeling. In another instance, a 26-in. log section was evacuated, then impregnated with sulfur dioxide gas until the digester registered a positive pressure of 10 psi. After being held in the sulfur dioxide atmosphere for 16 hr, the digester was vented and filled with water. After soaking for 30 min, the log section peeled satisfactorily. Hence, it appears that the process can be scaled up by properly adjusting the known variables. However, the data do not permit an economic appraisal.

To detect the possibility of damage from the unbuffered acid, 5.7 kg (d.b.) of chips from a sulfur dioxide debarked log were pulped by a standard kraft cook with the following results: screened yield = 56.6%; screenings = 0.4%; permanganate no. (25-ml basis) = 10; viscosity = 21.4 cp. These values compare favorably with the usual aspen pulps. Handsheet tests also gave usual values. In this instance debarking with acid imposed no penalty on either the pulp or paper.

LITERATURE CITED

1. Thimann, K. V., ed., "The Physiology of Forest Trees," p. 421, New York, The Ronald Press Co., 1958.
2. Crocker, W., ed., "The Growth of Plants," New York, Reinhold Publishing Co., 1948.
3. Browning, B. L., ed., "The Chemistry of Wood," New York, Interscience Publishers, 1963.
4. Newcomb, E. L., *Année Biologique* **31**: 397 (1955).
5. Isenberg, I. H. and Buchanan, M. A., *J. Forestry* **43**: 888–890 (1945).

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